CENTER
R and D LABORATORY
TECHNICAL REPORT

NO. 12662

IMPROVING THE HIGH TEMPERATURE
CREEP AND RUPTURE RESISTANCE
OF OXIDE-DISPERSION-STRENGTHENED ALLOYS

Department of the Army Project No. DAAE07-80-C-9091

by Case Western Reserve University
Cleveland, Ohio

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U.S. ARMY TANK-AUTOMOTIVE COMMAND
RESEARCH AND DEVELOPMENT CENTER
Warren, Michigan 48090

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PREFACE

The investigation reported upon herein was carried out in cooperation with the US Army Tank-Automotive Research and Development Command, Department of Defense, Warren, Michigan, under the terms of Contract DAAE07-80-C-9091. The Case Western Reserve University investigators, Professor L.J. Ebert and Mr. M.L. Telich, wish to acknowledge the support and assistance of Dr. James Chevalier, Chief, Armor and Components Function, and Mr. M.L. Holly of the Command. Mr. T. Wassel, formerly of the TARAD Command was very helpful in the initial stages of the investigation.

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The deformation annealing response of the ODS (Oxide-Dispersion-Strengthened) alloy MA 754 was examined in the effort to develop a thermomechanical technique whereby the alloy can be brought to near finish shaped components while at the same time improving the high temperature performance characteristics of the material. Major emphasis placed on 1) characterization of the as-received material in order to establish base - line properties, and 2) the evaluation of various thermomechanical processing schedules combining cold working (by rolling) and annealing in order to attain the most favorable grain morphology.

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The as-received alloy was found to be very amenable to cold rolling; initial experiments produced reductions on the order of 70%. Subsequent strain energy relaxation treatments however, were unsucessful in stabilizing the improved grain morphology produced by cold rolling. Utilization of a revised cold rolling plus anneal schedule (one involving step-wise cold working and annealing) was found to permit reductions on the order of at least 40% while still maintaining a stable large grained structure.

IMPROVING THE HIGH TEMPERATURE CREEP AND RUPTURE RESISTANCE OF OXIDE-DISPERSION STRENGTHENED ALLOYS

1.0 INTRODUCTION

The primary thrust of the research program discussed in this report is the development of processing techniques by which the best presently available high temperature alloys can be made to have performance characteristics superior to those which they presently possess. Another goal is to develop techniques for manufacturing components to near net shape by mechanical means, such as cold or warm forming, rather than by the presently used process of machining components from extruded bar stock. The latter process is wasteful of the costly alloys which are the basis of this study. One potential application for economically produced, high performance components is in gas turbine engines used in military and civilian applications.

The techniques employed in the study are those developed in the laboratories of the principal investigator, but which were not fully exploited on a practical basis in earlier investigations. The alloys which form the basis of the program likewise had been studied earlier in the laboratories of the principal investigator.

2.0 WORK EFFORT SUMMARY

2.1 Materials Selection

Two candidate materials were considered for this project, on the basis of previous studies. Both were ODS (Oxide Dispersion Strengthened) alloys. One, NiCrAlY, was considered because, in previous work, it was found to have the highest permissible operating temperature range of the commercially available alloys. The second material, known as MA 754*, was chosen for study under this project because of its more ready availability and its established high temperature data base.

When work was formally initiated, an order was placed for a billet of material 3.3 inches (83.8 mm) wide, 1.4 inches (35.6 mm) thick, and 21 inches (533.4 mm) long, from Huntington Alloys, Inc. A similar quantity of MA 754 was procured from Lewis Laboratory (NASA) in Cleveland, Ohio.

2.2 Materials Characterization

The material used in this project was the oxide dispersion strengthened nickel-base alloy MA 754 containing nominally 20% chromium, 0.3% aluminum, 0.4% titanium, and 0.6% yttria (Y_2O_3). The material was prepared by mechanical alloying, which involves the simultaneous milling of constituent powders of the above proportions with the eventual result of complete homogenization. The homogenized mixture is then "canned" in an evacuated steel

* MA 754 and NiCrAlY have similar compositions.

jacket, and hot-extruded into bars 3.3 inches (83.8 mm) wide, 1.4 inches (35.6 mm) thick, and 21 inches (533.4 mm) long (as delivered). Huntington Alloys, Inc., the manufacturer, provided the chemical analysis given in Table 1.

In order to obtain samples for initial characterization of the material (optical microscopic examination and subsequent hardness measurements), a test block was cut from the as-received bar. The test block length was approximately 3.5 inches (88.9 mm); the thickness and width dimensions were unchanged from that of the as-received bar. Figure 1 is a schematic showing the location of the samples relative to the extrusion direction.

Standard metallographic polishing techniques were used for sample preparation. They consisted of: 1) rough polishing with a series of SiC papers, 240 grit thru 600 grit; 2) final polishing involving the use of diamond paste impregnated texmet wheels followed by lapping on a 0.3μ Al_2O_3 impregnated wheel.

Satisfactory grain boundary delineation was accomplished by the use of chemical etch consisting of 5 parts HCl (conc), 2 parts HNO_3 (conc), and 2 parts methanol (95%). Acid staining of the surface was greatly reduced by swabbing the etch onto the surface instead of immersing the specimens. Microstructural examination of the specimens in Figure 1 revealed no significant variation either in grain size, shape, or in the distribution of the dispersed phase across the bar. Optical photomicrographs of typical longitudinal and transverse sections are provided in Figure 2.

Table 1. Vendor's Chemical Analysis of MA 754 Bar Heat #DT0123B

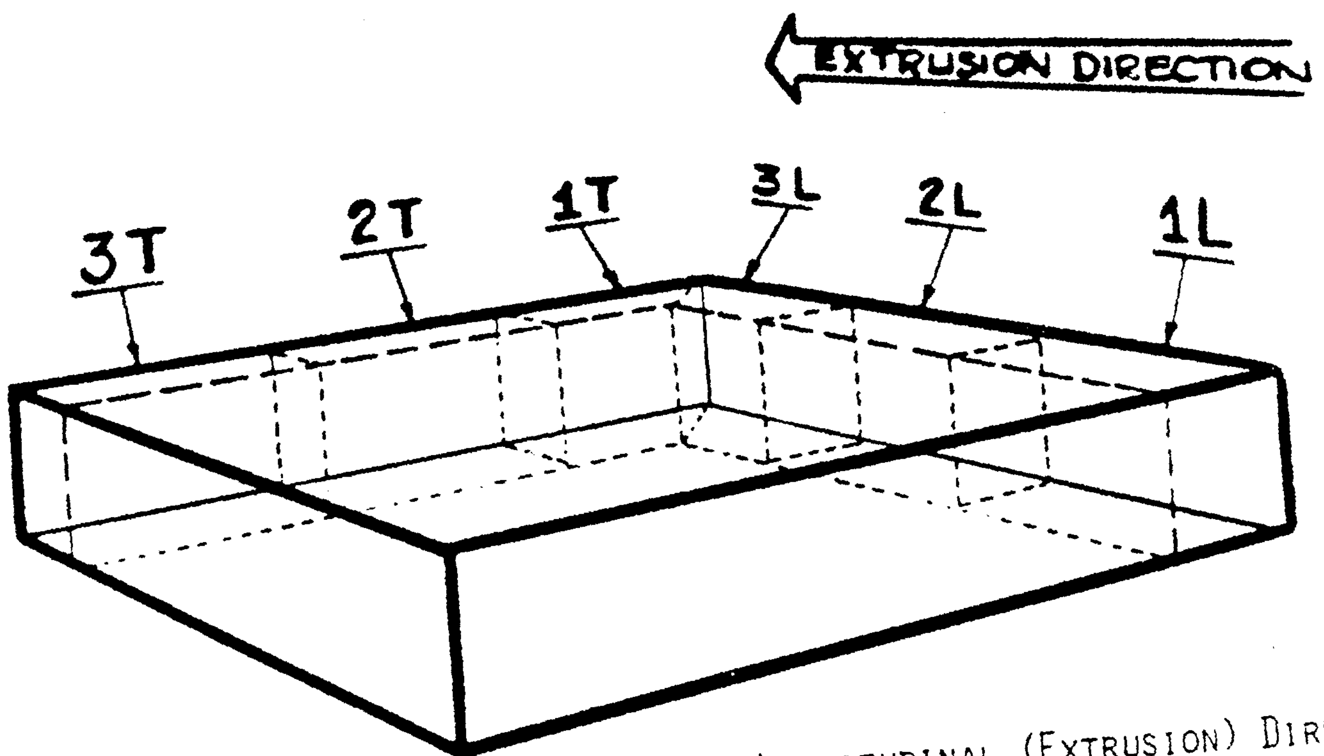
ELEMENT	% (By Weight)
C	0.06
Fe	0.93
S	0.002
Ni	77.20
Cr	20.15
Al	0.31
Ti	0.41
O	0.33
Y_2O_3	0.61

TEST BLOCK DIMENSIONS

Length ------ 3.5" (88.9 mm)

Width ------- 3.3" (83.8 mm)

Thickness --- 1.4" (35.6 mm)



L= LONGITUDINAL (EXTRUSION) DIRECTION

T= TRANSVERSE DIRECTION

Figure 1: Location of Test Samples

Quantitative grain dimension measurements were made by the line-intercept method, using at least 35 test lines for each of the three orthogonal directions. The basic quantity measured in the line-intercept method is N, the number of grain boundary intersections per length of test line. The linear grain dimension L is defined at the inverse of N. By measurements of L in three orthogonal directions, it was possible to estimate an average grain diameter by the following relation:

$$L_{AVE} = 0.344\ (L_1 L_2 L_3)^{1/3}$$

where L_{AVE} = average grain diameter

L_1 = grain diameter in the longitudinal (extrusion) direction

L_2 = grain diameter, long transverse (width) direction

L_3 = grain diameter, short transverse (thickness) direction

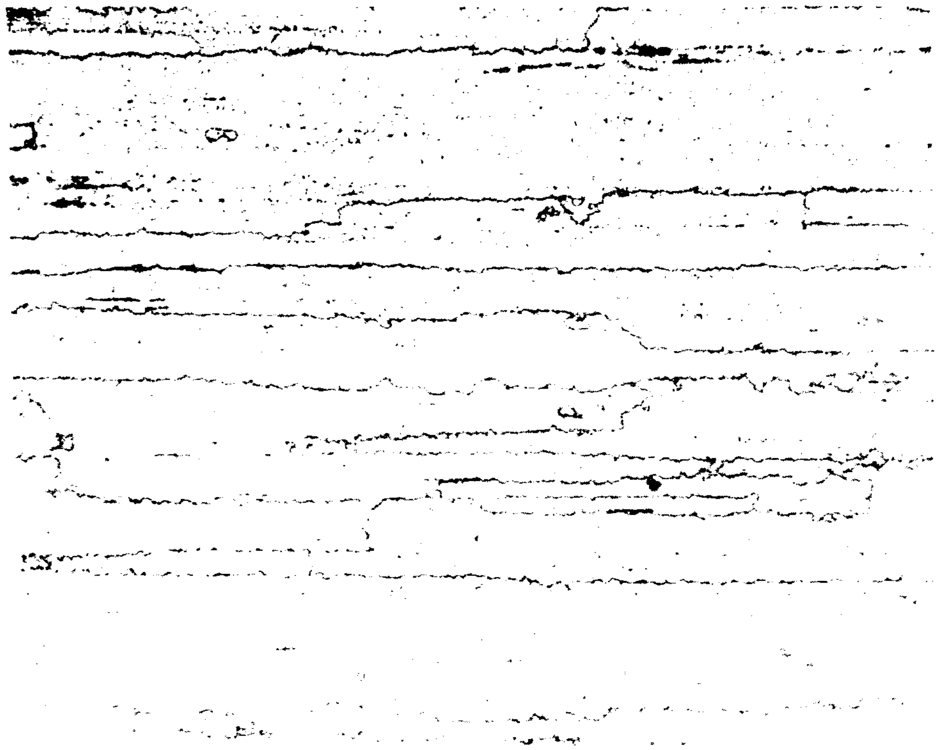
Since it was readily apparent that the material is not equiaxed, grain shape measurements were made. Grain shape is defined as the ratio of the length to width of the individual grains or L/D, and is computed from the linear grain dimension measurements described earlier:

$$L/D = 2L_1/(L_2+L_3)$$

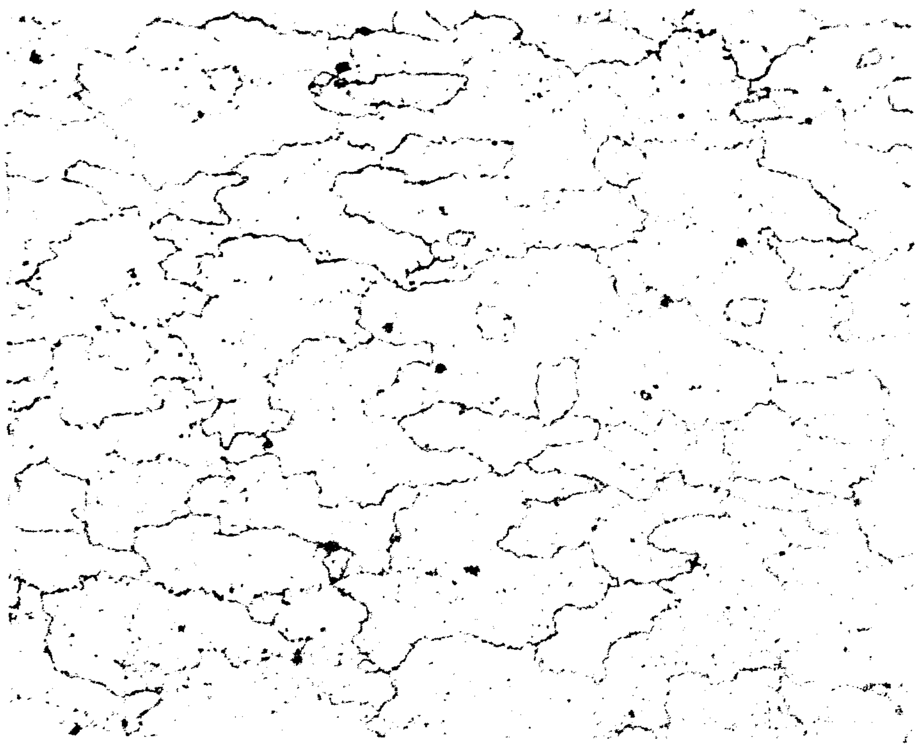
where L/D = length to width ratio

L_1, L_2, L_3 = grain diameters as defined above.

The results of the grain size and shape calculations are given in Table 2. Note that the relatively large grain diameter



a. Longitudinal (Extrusion) Direction



b. Transverse (Cross-Extrusion) Direction

Figure 2: Optical Photomicrographs of MA 754 Alloy.
100 X Magnification

(0.17 mm) and large L/D ratio (approximately 7) eliminate the need for thermal or mechanical treatments, prior to mechanical testing or cold working, to achieve the desired grain size and aspect ratio.

As a further quantitative evaluation of the as-received material, the Rockwell "C" (R_C) hardness was determined. By using the metallographic specimens shown in Figure 1, a complete hardness profile of the block was determined (Figure 3). The profile reveals no significant variation in hardness with direction or distance; this seems to support the observation made earlier regarding the uniformity of grain size, shape, and distribution of the dispersed phase. Table 3 lists the average R_c hardness values for each specimen in addition to the overall hardness value.

On the basis of the above evaluations, the material was judged to be a good representative sample of its alloy class.

2.3 Elevated Temperature Tensile Properties - as-received material

2.3.1 Longitudinal (Extrusion) Direction

In order to assess the material's basic strength and ductility at elevated temperatures as well as to evaluate its cold (or warm) working potential, tensile tests (in duplicate or triplicate) were conducted on MA 754 test specimens at temperatures ranging from ambient to 2000°F (1093°C).

Table 2: Grain Size and Shape Measurements

DIRECTION	L (mm)
LONGITUDINAL (EXTRUSION)	0.7886
LONG TRANSVERSE (WIDTH)	0.1602
SHORT TRANSVERSE (THICKNESS)	0.0678

L_{AVE} = 0.1727 mm

L/D = 6.917

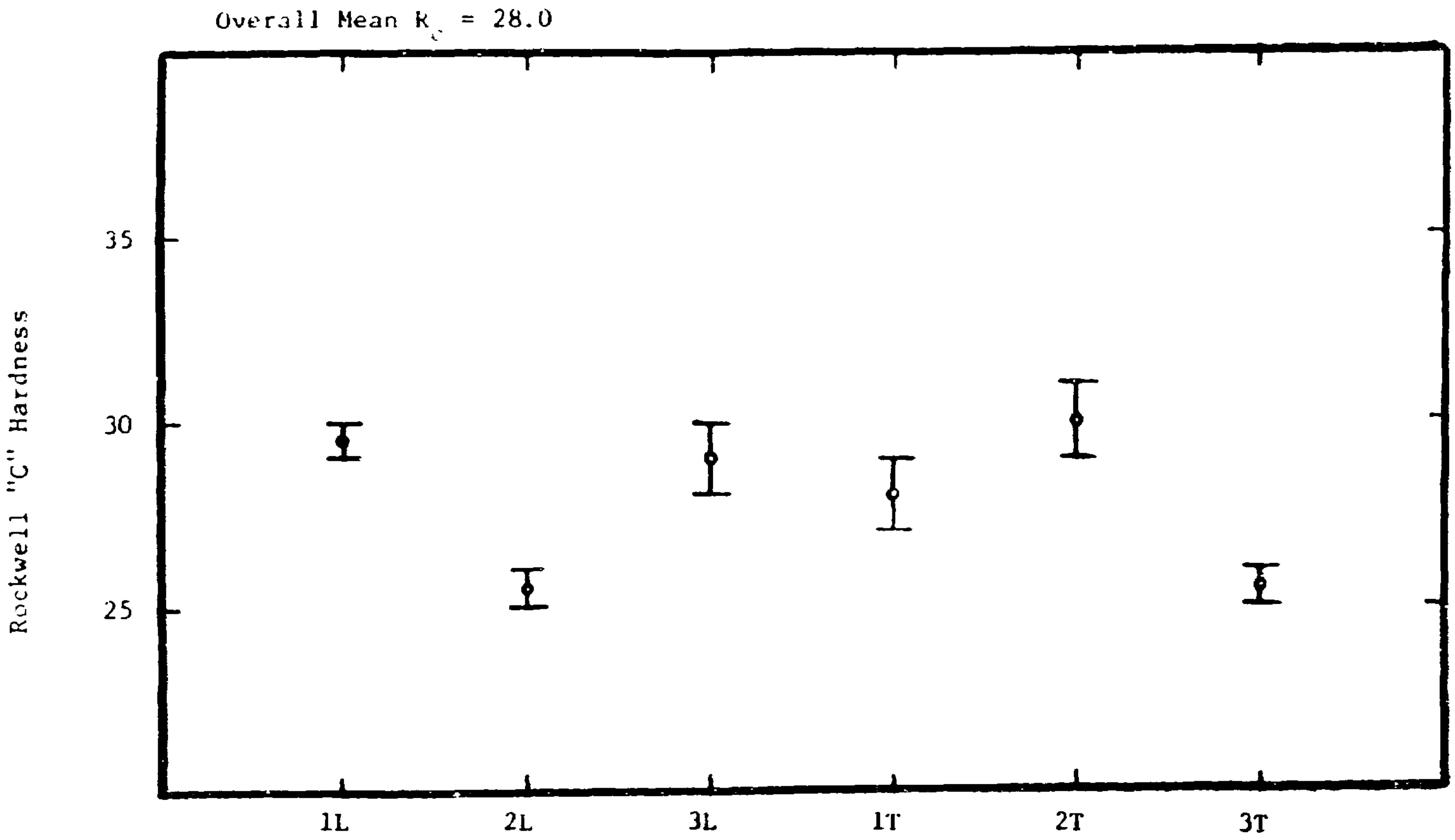


Figure 3: Hardness Profile of Test Block Removed From As-Received MA 754 Alloy Bar.

Table 3: Average Rockwell "C" Hardness Values of Specimens Cut from Test Block #1

SPECIMEN	HARDNESS - R_c
1L	29.5
2L	25.5
3L	29.0
1T	28.0
2T	30.0
3T	25.5

OVERALL AVERAGE R_c = 28.0

The specimen design used in the tensile tests is shown in Figure 4. The button-head gripping feature was incorporated into the design to maximize loading axiality. The specimen grips used in testing were made of MAR M246 and were designed to undergo no deformation. A boron nitride powder was used to prevent diffusion bonding between the specimen heads and grips.

The test apparatus used to perform the tensile tests was an Instron-Satec furnace combination. Temperature was controlled to ±2°F (±1°C) over the entire gage length of the specimen, using Chromel-Alumel thermocouples for temperature sensing.

The results of the tensile tests in the extrusion direction of the MA 754 alloy are shown in Figures 5 and 6. It is significant to note that the material is very ductile at ambient temperature and that it retains good strength and ductility at 2000°F, indicating high useable load carrying capability and toughness at this temperature.

Microscopic examination of longitudinal tensile piece fractures revealed two primary failure modes. At temperatures below 1000°F (538°C), transgranular failure is readily apparent (Figure 7). Above 1000°F intergranular failure, accompanied by extensive cavitation, is observed (Figure 8).

An important feature of the intergranular mode is the locus of failure. Figure 9 is an optical photomicrograph of a longitudinal test piece section far from the fracture surface. Note that the intergranular cracking characteristic of this failure mode occurs only at the transverse grain boundaries. It is apparent that any effort to improve the high temperature performance of MA 754 should include a reduction in the number of transverse grain boundaries.

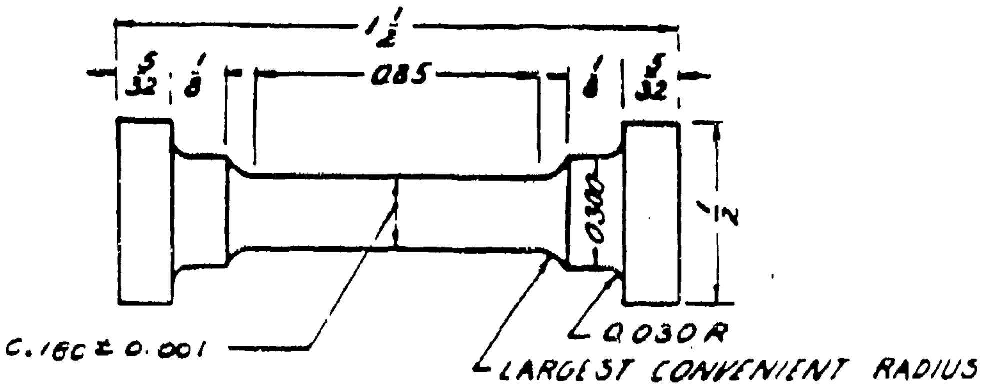


Figure 4: Specimen Design Used in Determining the Tensile Properties of As-Received MA 754.

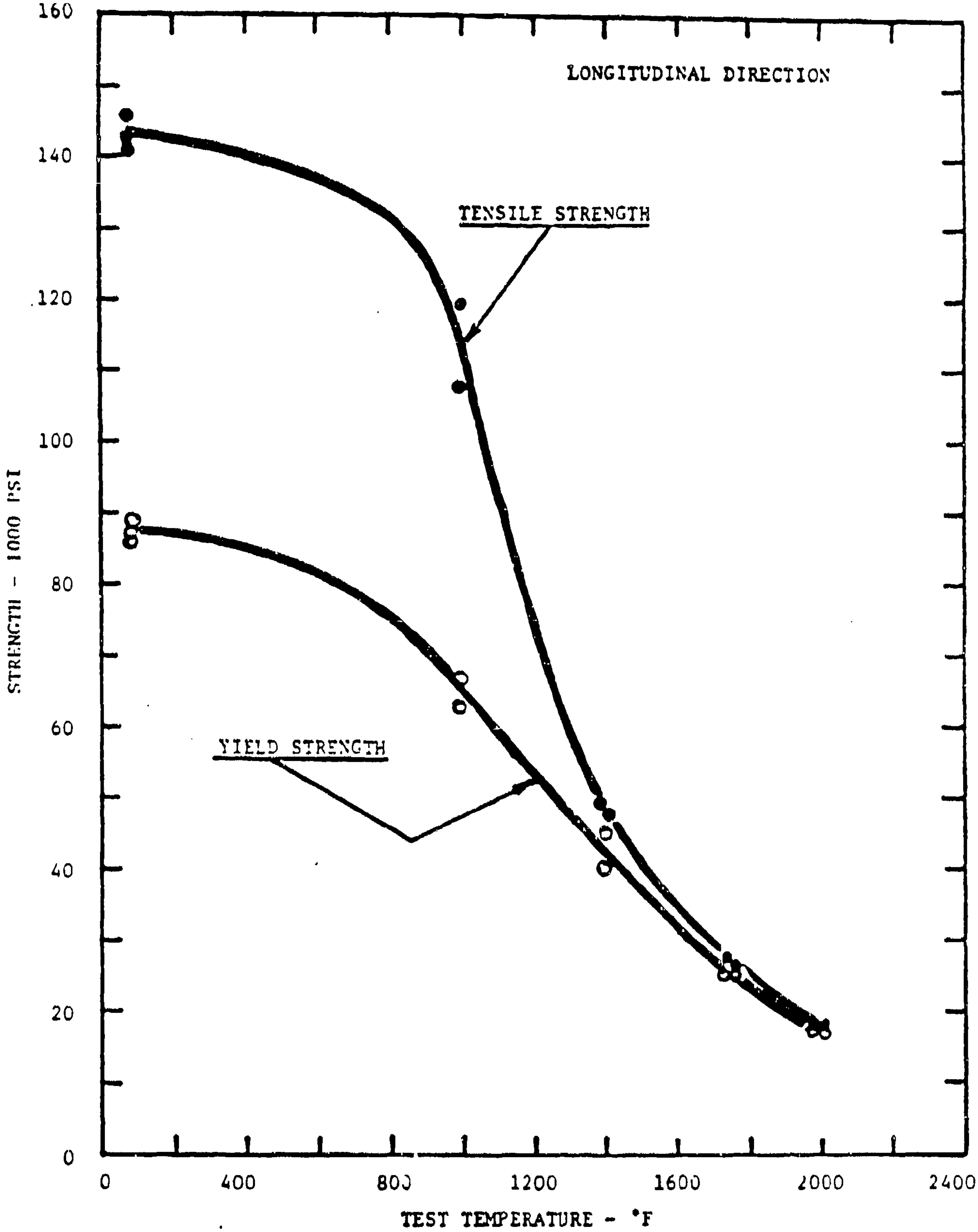


Figure 5: Tensile and Yield Strength vs Temperature for MA 754 Alloy Tested in the Longitudinal (Extrusion) Direction.

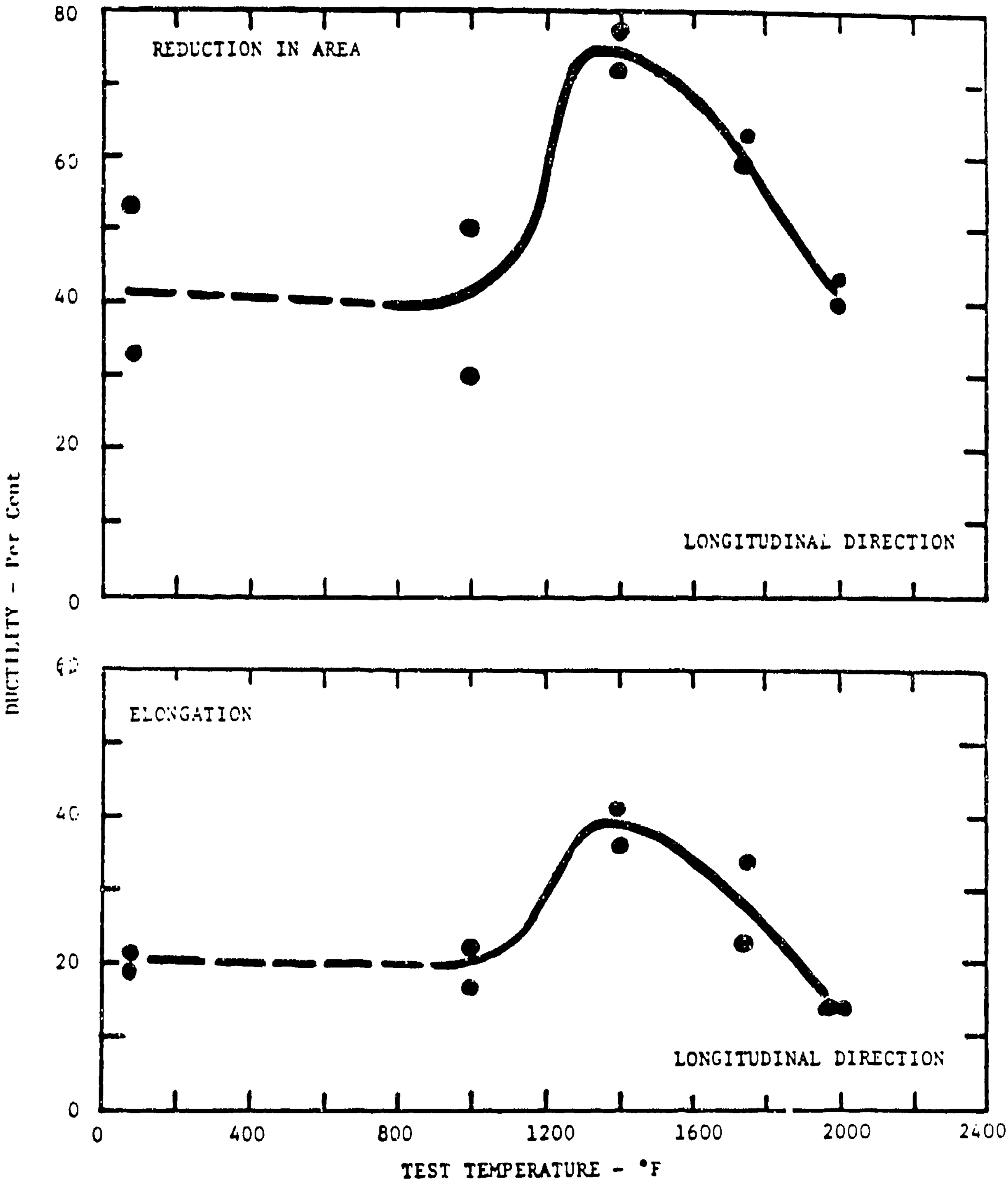


Figure 6: Reduction in Area (Top) and Elongation (Bottom) vs Temperature for MA 754 Tested in the Longitudinal Direction.

Figure 7: Optical Photomicrograph of the Mid-Section of a Longitudinal Tensile Test Piece, Showing Fracture Edge. Test Conducted at Room Temperature.
100 X Magnification
Note the transgranular fracture mode.

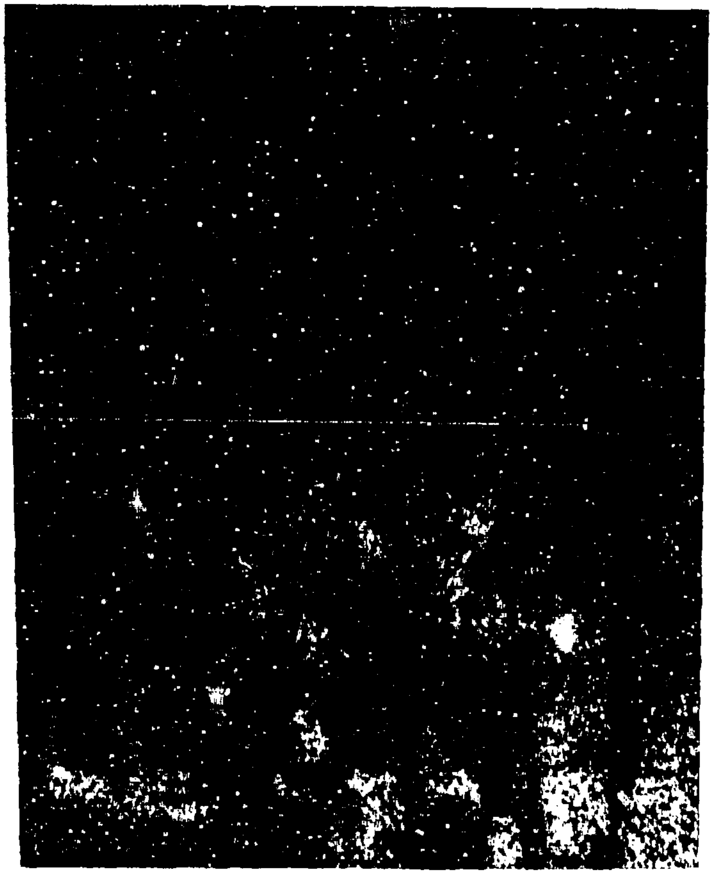


Figure 8: Optical Photomicrograph of the Mid-Section of a Longitudinal Tensile Test Piece, Showing Fracture Edge. Test Conducted at 2000°F (1093°C). 100 X Magnification
Note the intergranular nature of the fracture, and the extensive cavitation.

Figure 9: Optical Photomicrograph of the Midplane of a Longitudinal Tensile Test Piece Remote from the Fracture. Test Conducted at 1750°F (954°C). 100 X Magnification
Note the intergranular cracks at the transverse grain boundaries.

2.3.2 Transverse Direction

While it is often possible to design components so that the major loading axis is coincident with the strong material direction of the component, in many cases the "transverse" properties play a significant role. Hence, a limited study has been done to assess the transverse properties of MA 754. The results are shown in Figures 10 and 11.

Microstructural examination of fractured transverse tensile test specimens reveal a situation quite similar to that found in fractured longitudinal test specimens. Below 1000°F (527°C), transgranular failure predominates (Figure 12), while above 1000°F intergranular failure is apparent with extensive cracking at transverse grain boundaries (Figure 13).

2.4 Elevated Temperature Creep Properties - As-Received Material

All creep testing was done utilizing leveled creep racks (12,000 lb. capacity) modified to produce constant stress rather than constant load. The furnaces were of the Satec tube-type, with a maximum temperature at 2200°F (1204°C) and an accuracy in the hot zone of ± 2°F (± 1°C). Strains were measured with a dial indicator with an accuracy of 0.0001 inch (0.0025 mm) or an LVDT* with an accuracy of ± 0.00005 inch (0.0013 mm). The test piece design used in the determination of the creep properties of the as-received material was basically the same as that used to determine the tensile properties and is shown in Figure 14.

* Linearly Variable Differential Transformer

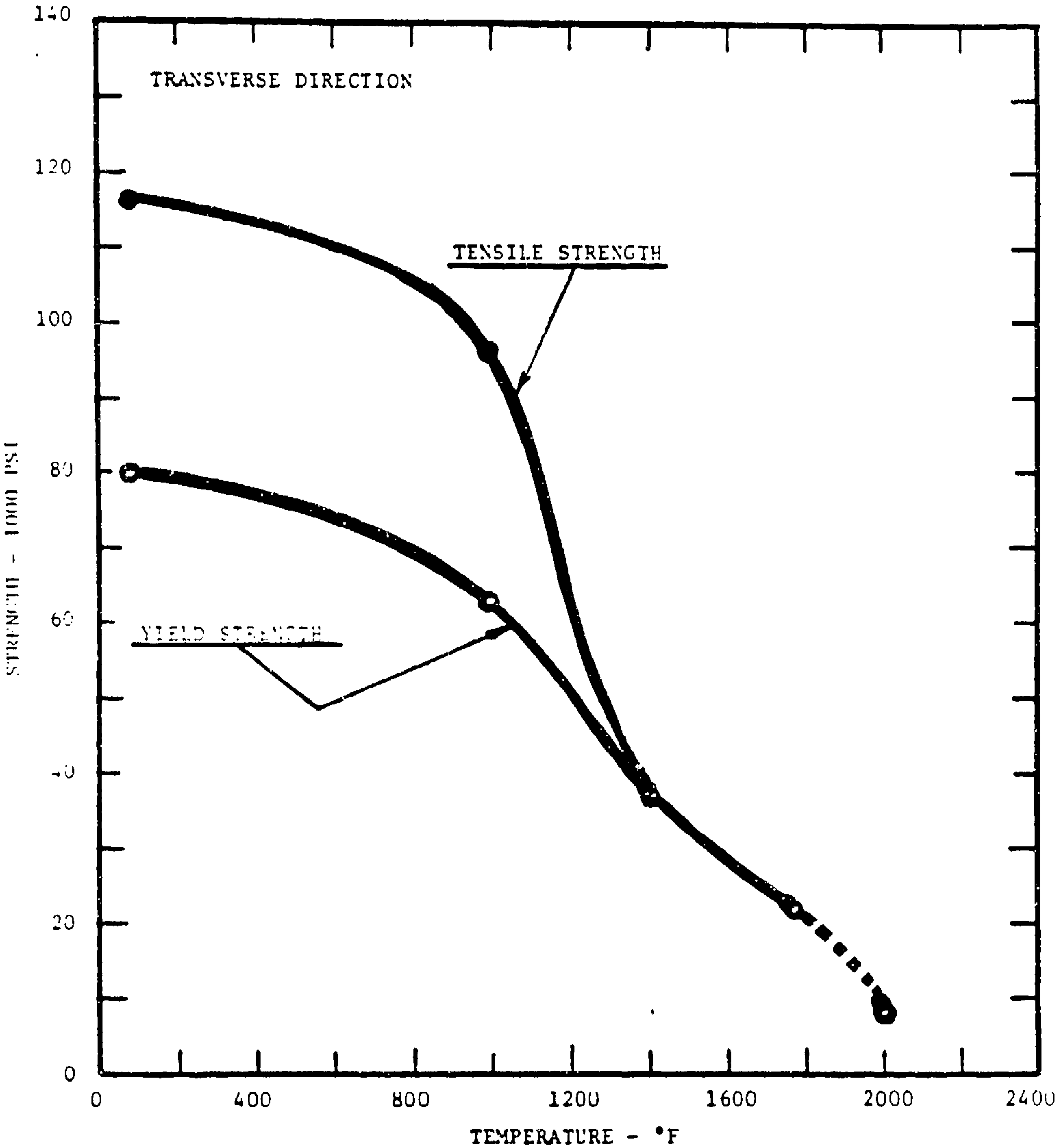


Figure 10: Tensile and Yield Strength as a Function of Test Temperature for MA 754 Tested in the Transverse (Cross-Extrusion) Direction.

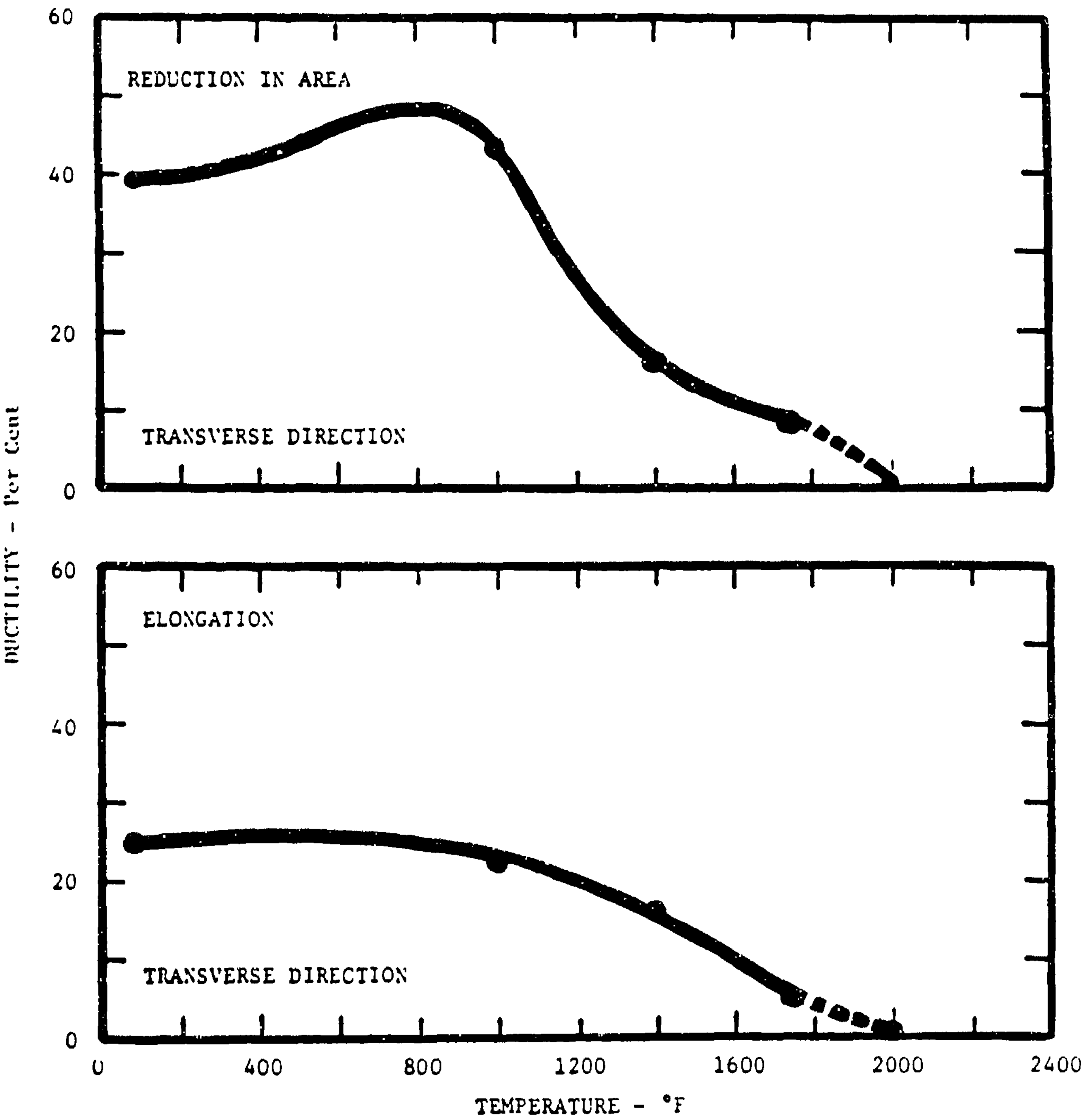


Figure 11: Reduction in Area (Top) and Elongation (Bottom) as a Function of Test Temperature for MA 754 Tested in the Transverse Direction.

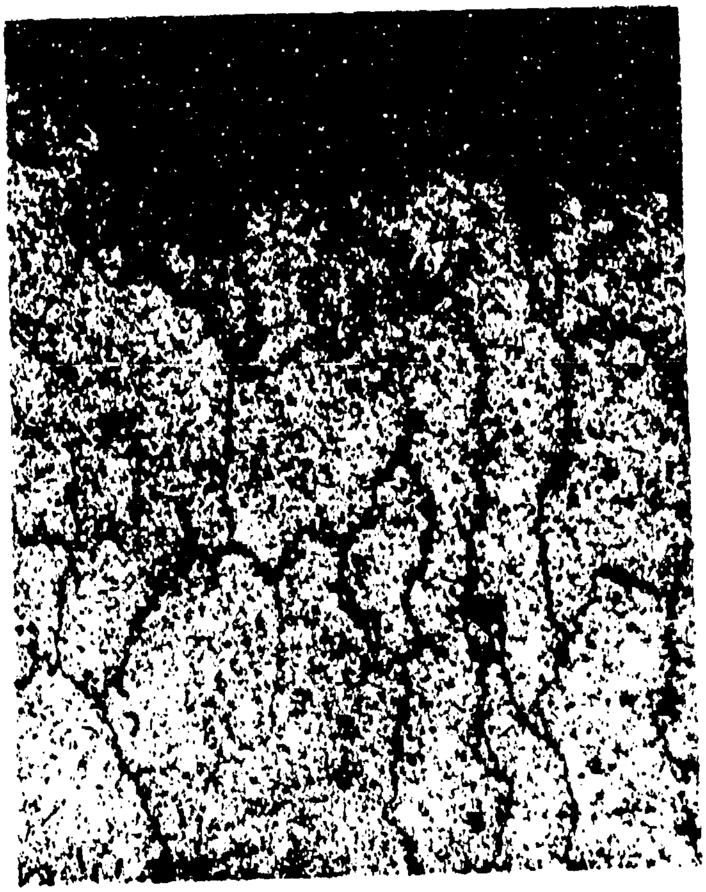


Figure 12: Optical Photomicrograph of the Midplane of a Transverse Tensile Test Piece Showing Fracture Edge. Test Conducted at 1000°F (537°C). 200 X Magnification
Note the transgranular fracture mode.

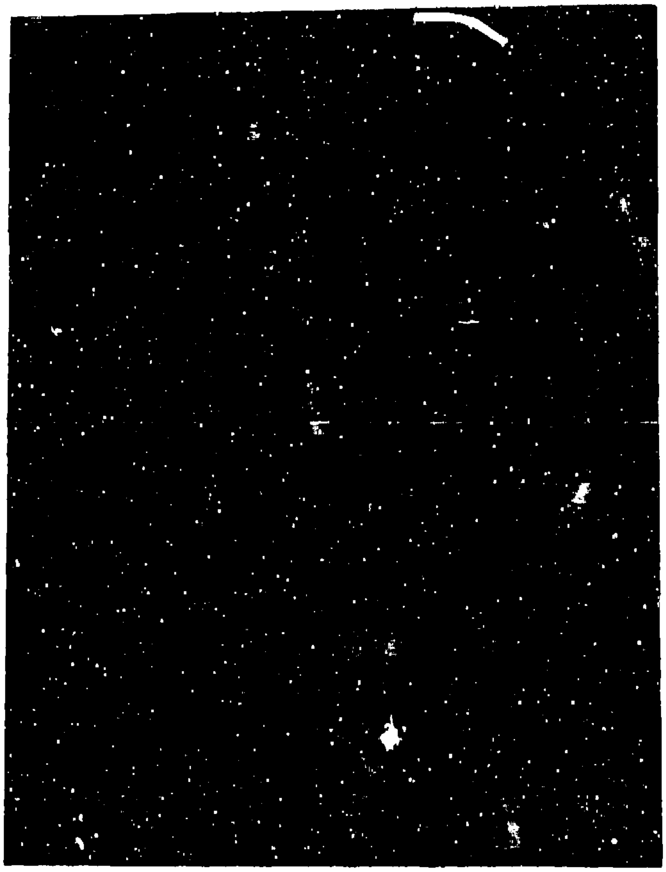


Figure 13: Optical Photomicrograph of the Midplane of a Transverse Tensile Test Piece, Showing Fracture Edge. Test Conducted at 1400°F (760°C). (200 X Magnification)
Note the intergranular fracture mode, and the extensive cavitation.

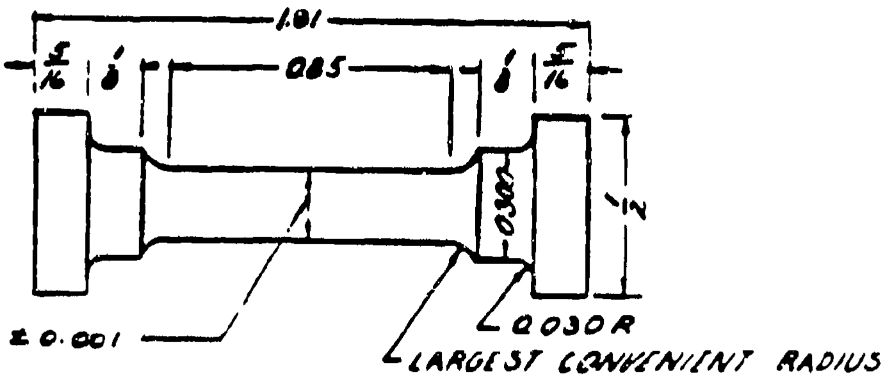


Figure 14: Specimen Design Used in Determining the Creep Properties of As-Received MA 754.

The quantities of primary interest measured during creep tests in the present study were: 1) steady state creep rate, $\dot{\varepsilon}_s$; 2) time to failure (rupture time) t_r; 3) elongation at time of rupture; and 4) reduction in area at time of rupture. These were determined at two different temperatures, 1600°F (871°C) and 1800°F (982°C), with four stress levels at each temperature. Tables 4 and 5 are a summary of all creep test results conducted with the as-received material.

In addition, Figures 15 and 16 reveal typical creep curves for each of the temperatures studied.

Figures 17 and 18 show the steady state creep rate as a function of applied stress at the two temperatures studied. The stress dependence of MA 754 in creep is quantified by the stress exponent, n, which is related to the steady state creep rate and the applied stress by the following relation:

$$\dot{\varepsilon}_s \sigma_a^{\ n} T = \text{const}$$

By plotting ln $\dot{\varepsilon}_s$ versus lnσ_a, the stress exponent, n, can be determined as the slope of the curve.

According to Figures 17 and 18, the stress exponent of MA 754 at 1600°F (871°C) is 19.6, and at 1800°F (982°C) is 28.2. As a result, it would appear that the stress dependence is not independent of temperature. However, these values are consistent with the high stress dependence of the steady state creep rate observed for ODS materials.

At this time, no conclusions are warranted concerning the creep activation enthalpy of the as-received MA 754. More data must be generated to formalize such conclusions.

Table 4: Creep Properties of MA 754 Alloy at 1600°F (871°C)

STRESS LEVEL (FRACTION OF TENSILE STRENGTH AT 1600°F)*	MINIMUM CREEP RATE ($\dot{\varepsilon}$/sec)	RUPTURE TIME (HRS)	RUPTURE ELONGATION (%)	RUPTURE AREA RED (%)	TOTAL TIME (NON-RUPTURE) (HRS)**
0.57	1.48×10^{-9}	----	----	----	238
0.65	7.94×10^{-9}	----	----	----	253
0.70	4.72×10^{-8}	347	11	22.5	----
0.75	4.38×10^{-7}	41.5	14	40	----

* Longitudinal Tensile Strength at 1600°F is ~ 35,000 PSI (241.3 MPa)

** Tests Interrupted Prior to Fracture

Table 5: Creep Properties of MA 754 at 1800°F (982°C)

STRESS LEVEL (FRACTURE OF TENSILE STRENGTH AT 1800°F)*	MINIMUM CREEP RATE ($\dot{\epsilon}$/sec)	RUPTURE TIME (HRS)	RUPTURE ELONGATION (%)	RUPTURE AREA RED (%)	TOTAL TIME (NON-RUPTURE) (HRS)**
0.75	5.19×10^{-9}	-----	-----	-----	338
0.80	3.77×10^{-8}	205.5	8.0	18.8	-----
0.85	1.55×10^{-7}	70.0	10.0	20.4	-----
0.90	9.83×10^{-7}	13.7	12.6	29.7	-----

* Longitudinal Tensile Strength at 1800°F is $\sim$ 25,000 PSI (172.4 MPa)

** Tests Interrupted Prior to Fracture

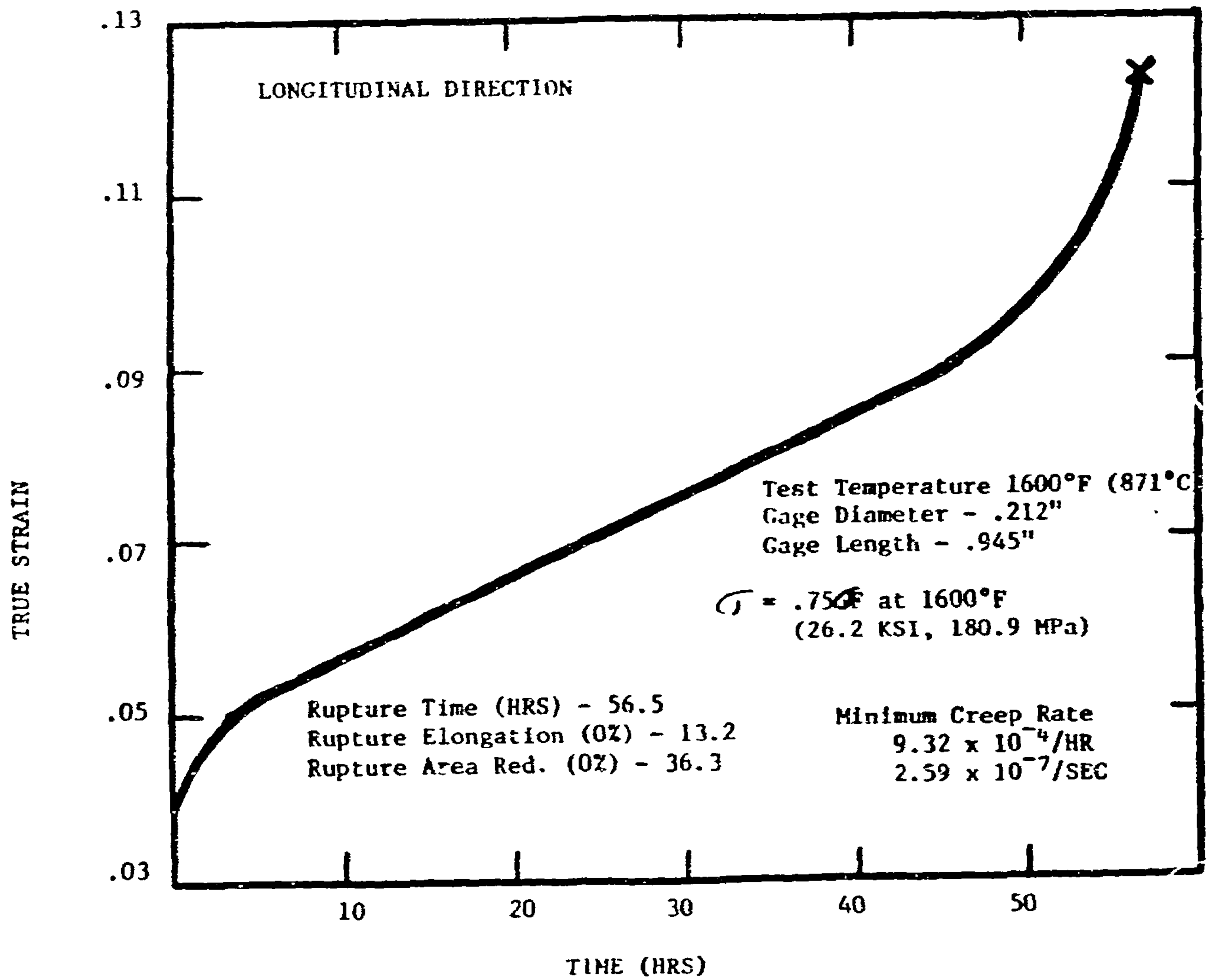


Figure 15: Creep-Rupture Curve for MA 754 Tested at 1600°F (871°C).

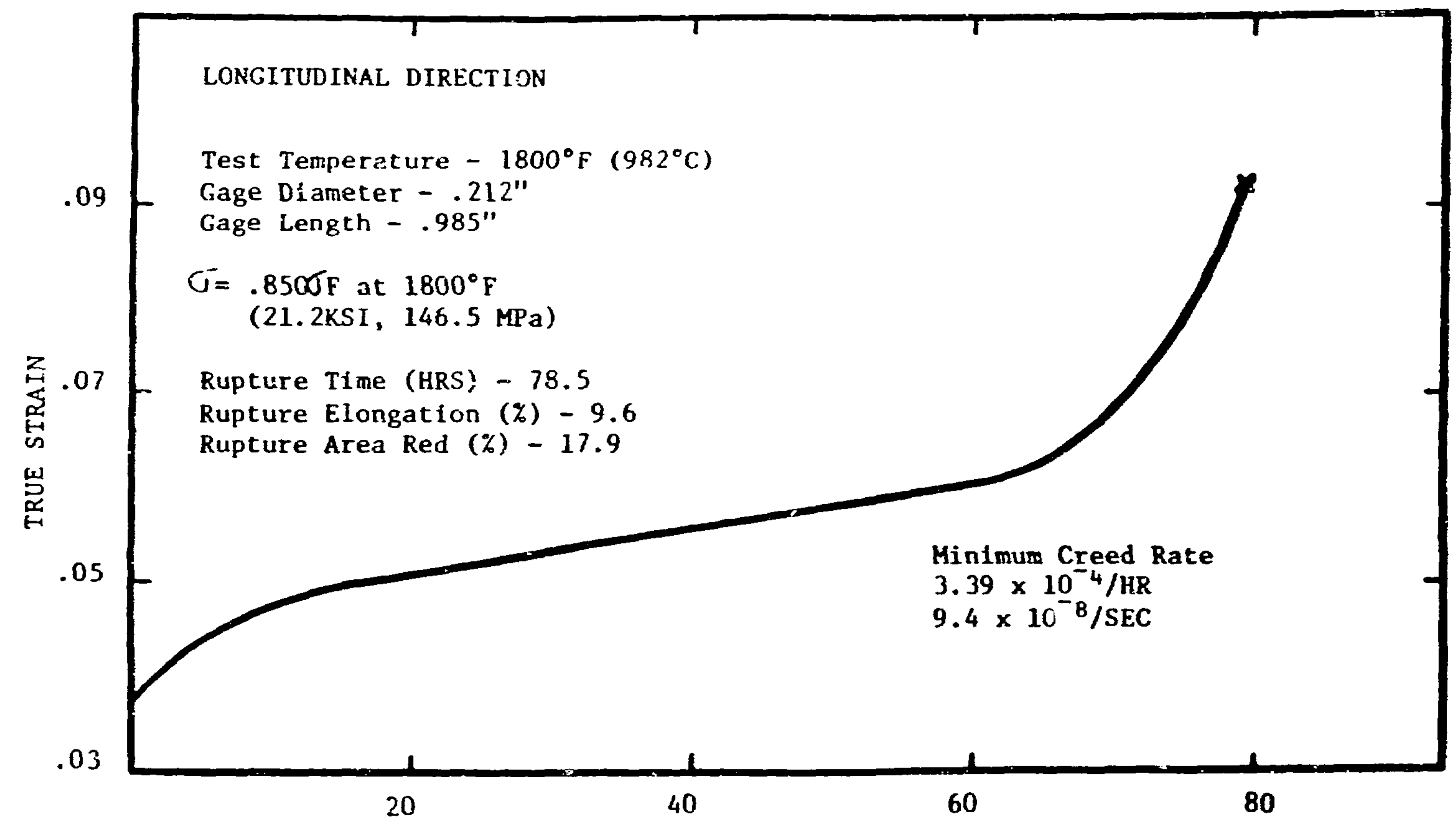


Figure 16: Creep-Rupture Curve for MA 754 Tested at 1800°F (982°C).

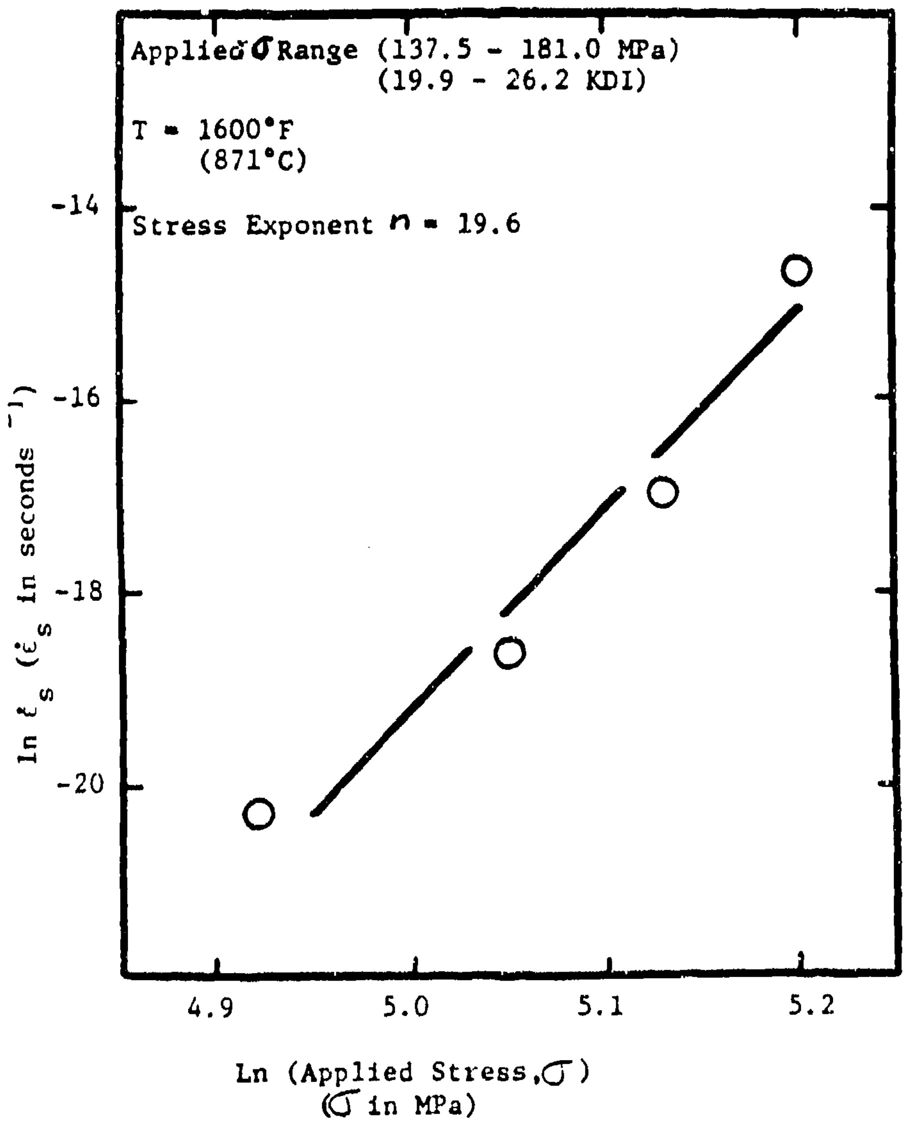


Figure 17: Ln (Steady State Creep Rate) vs Ln (Applied Stress) for As-Received MA 754 Alloy Tested in Creep at 1600°F (871°C).

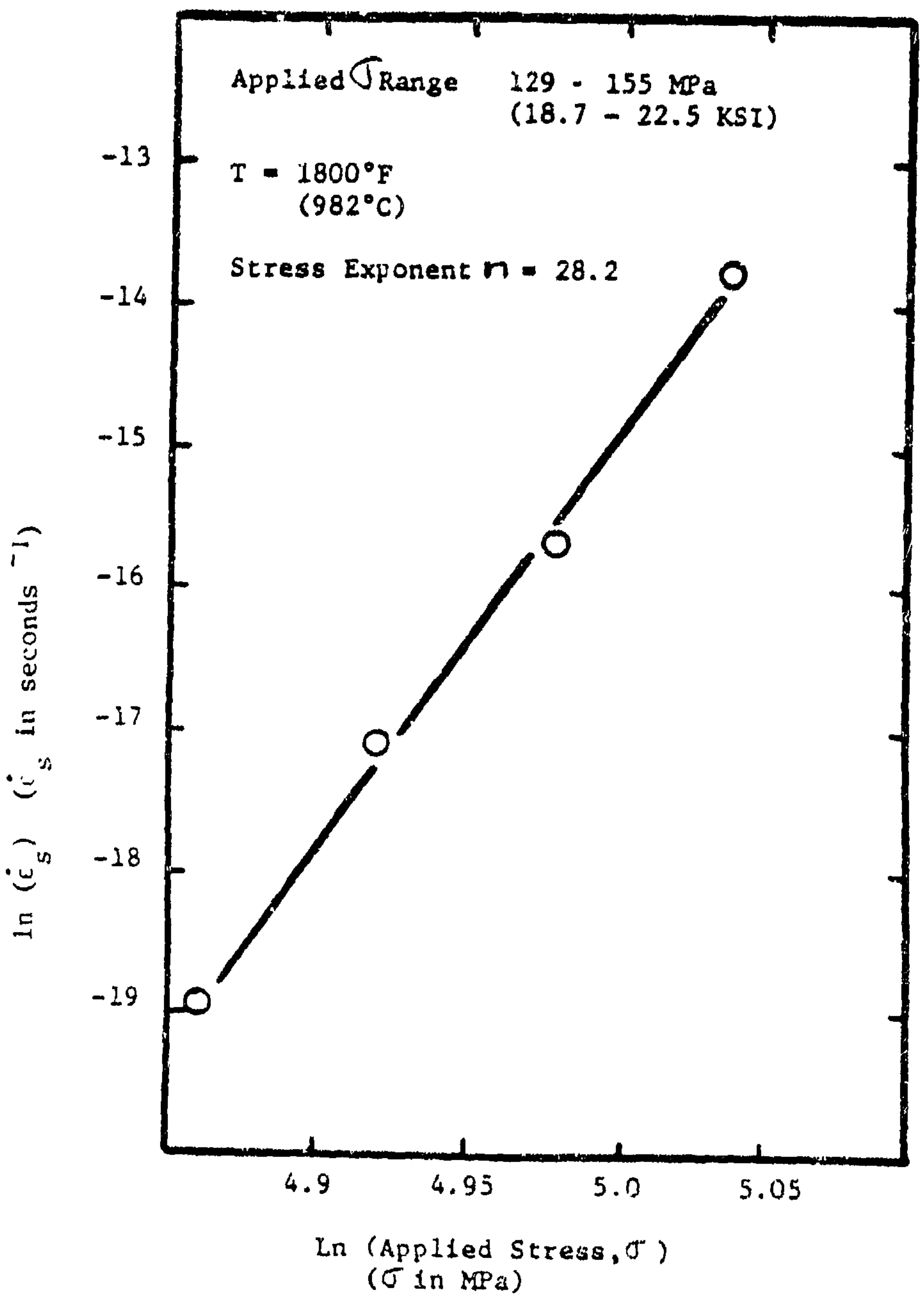


Figure 18: Ln (Steady State Creep Rate) vs Ln (Applied Stress) for As-Received MA 754 Alloy Tested in Creep at 1800°F (982°C).

As was the case with specimens ruptured during tensile tests, effort has been expended on examining the microstructure of the fracture area of creep-tested specimens.

Metallography of failed specimens of MA 754 revealed that fracture is intergranular at the temperatures studied. It is important to note that this failure mode is the same as that observed upon microstructural examination of longitudinal tensile specimen fractures.

An important feature of the intergranular mode is the locus of failure. Figure 19 is an optical photomicrograph of a creep specimen subjected to a test which was interrupted in the steady state region after 4.8% elongation revealing void formation at transverse grain boundaries. As the test proceeds into third stage creep, the voids coalesce and enlarge, eventually resulting in fracture. This intergranular damage is seen in all crept material.

2.5 Ambient Temperature Deformation and Annealing Response of MA 754 Alloy

As stated earlier, the primary purpose of this research program was development of techniques by which MA 754 can be processed into near-finish shaped components by mechanical means, while at the same time increasing the permissible operating temperatures of the components. In other words, one of the intents of the study was that of developing a technique whereby the alloy can be cold rolled to useful reductions, while at the same time maintaining a favorable grain morphology.

2.5.1 Cold Rolling of MA 754 to 70% Reduction in Thickness

2.5.1.1 Characterization

In order to find the degree to which the ODS alloy MA 754 was amenable to ambient temperature deformation for pre-form purposes, rolling tests were performed on a blank of the as-received material. The blank was initially 3.36 x 1.05 x 0.347 inches (85.3 mm x 26.7 mm x 8.8 mm) in dimensions and found to be amenable to rolling to a thickness reduction of some 70%, finishing with the approximate dimensions of 10.5 x 1.07 x 0.106 inches (266.7 mm x 27.2 mm x 2.7mm), with no intermediate anneals. The cold rolling was halted at 70% reduction so that the rolled piece was thick enough for subsequent annealing processing. It appeared that the material was capable of even more cold rolling reduction, because at the time the rolling was stopped, there were no evidence of edge or side cracking of the piece.

Cold rolling of the MA 754 blank greatly improved the aspect ratio of the long grains, and produced flattening in the short transverse direction. Figure 20 shows the longitudinal and transverse microstructures of the rolled material.

2.5.1.2 Recrystallization Response

As a result of cold rolling the as-received alloy to 70% reduction in thickness, it became necessary to attempt the development of an appropriate strain energy relaxation treatment in order to stabilize the improved grain morphology shown in Figure 20.

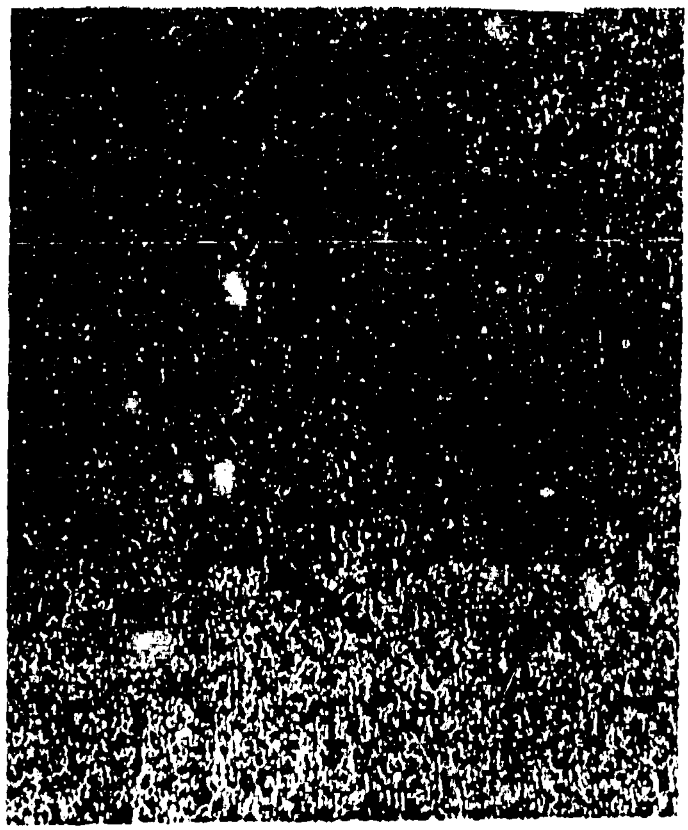
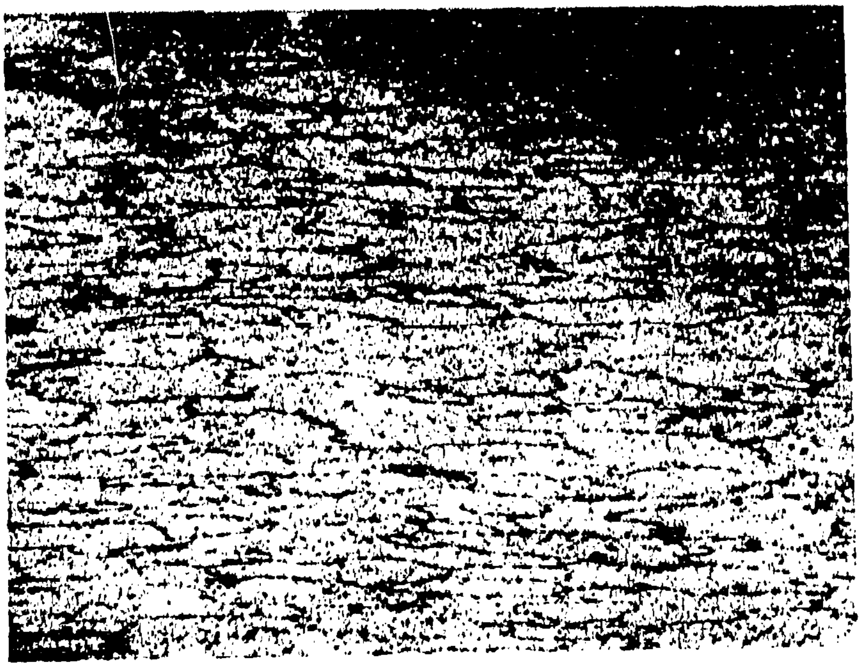


Figure 19: Optical Photomicrograph of the Mid-Section of a Longitudinal Creep Test Piece, Showing Void Formation at Transverse Grain Boundaries. Creep Test Interrupted Prior to Failure, with Creep Strain being = 0.048.
Test Temperature 1600°F (871°C)
Applied Stress 22,700 Psi (156.8 MPa)
Specimen Etched; 320 X Magnification

a. Longitudinal Section



b. Transverse Section

Figure 20: Optical Photomicrographs of MA 754 Alloy Cold Rolled to 70% Reduction in Thickness.
Specimen Etched; 100 X Magnification.

As a prelude to development of the treatment, the recrystallization response of 70% cold rolled MA 754 was determined from nine separate anneal experiments. Specimens were machined from the as-rolled blank, and annealed for four hours at specified temperatures from 1000°F to 2400°F (538°C to 1315°C). The anneals were conducted utilizing a standard tube furnace with a dried argon atmosphere to prevent oxidation. The temperature was controlled to ± 3°F (± 1.7°C).

Figure 21 is the recrystallization curve for the cold rolled material. The annealing temperature at which fine, equiaxed grains were first noted was 1200°F (649°C), corresponding to an approximately 5% drop in hardness as compared to the as-rolled material. This temperature is important because of the possible actions of strain relaxation mechanisms prior to recrystallization. At 1300°F (704°C), the average Rockwell "C" hardness was 34.5, a significant reduction in the as-rolled hardness value. Figure 22 is an optical photomicrograph revealing the recrystallized grain structure of the specimen annealed at 1300°F (704°C). The complete list of hardness values for each of the annealed specimens is provided in Table 6.

2.5.1.3 Intermediate Annealing Response

In order to test the effectiveness of the possible action of strain relaxation mechanisms in preventing the recrystallization of MA 754 cold rolled 70%, the following experiments were performed:

As Rec'd MA 754 Cold Rolled 70% + Anneal at 1200°F, four hours + Anneal at 2400°F, four hours, (in argon)

As Rec'd MA 754 Cold Rolled 70% + Anneals at 1100°F, 1200°F, and 1300°F, four hours each + Anneal at 2400°F, four hours (in argon)

As Rec'd MA 754 Cold Rolled 70% + Anneal at 2400°F, four hours (in argon).

Results of these experiments indicated that all three samples recrystallized completely to a generally equiaxed, fine grain structure. (Figure 23).

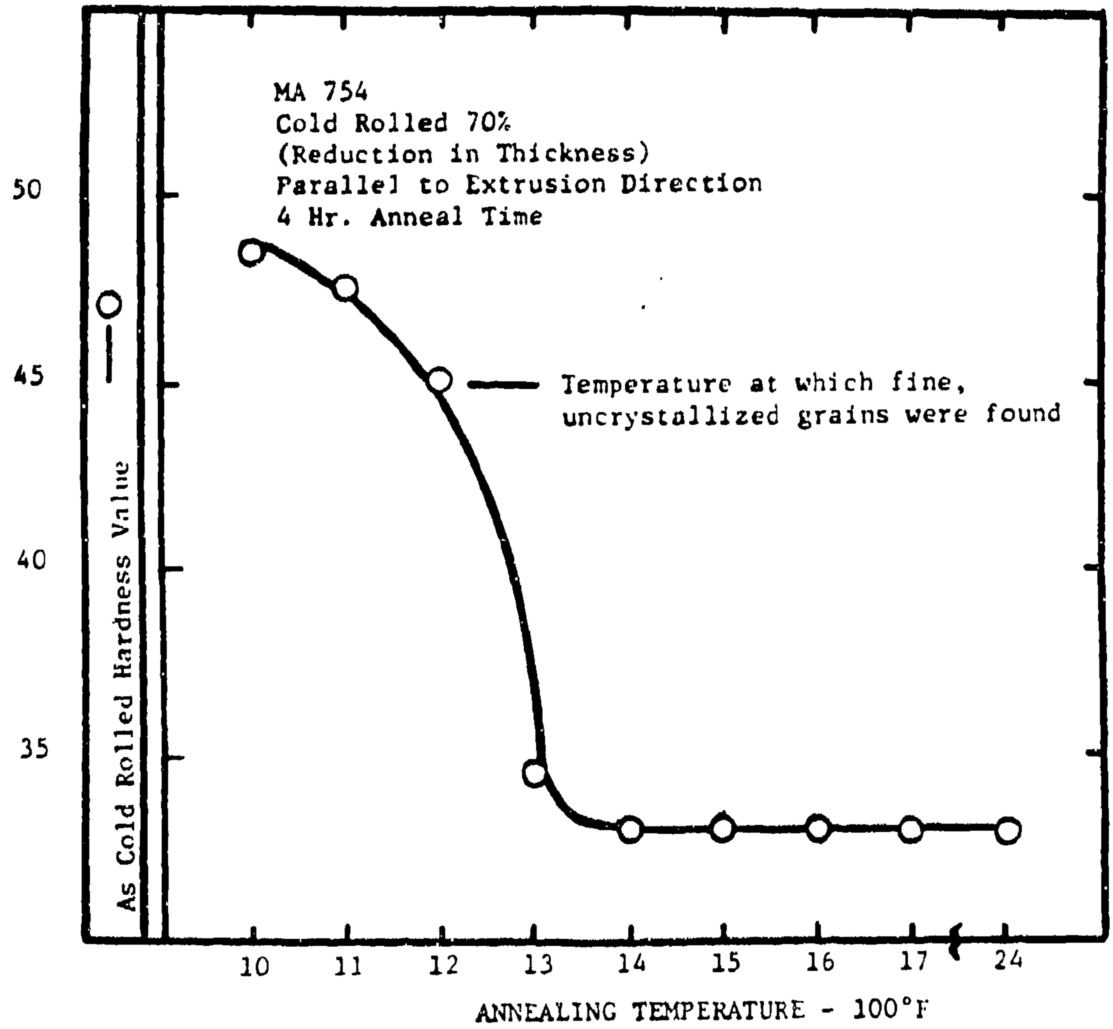


Figure 21: Recrystallization Curve for MA 754 Cold Rolled Parallel to the Extrusion Direction, to 70% Reduction in Thickness.

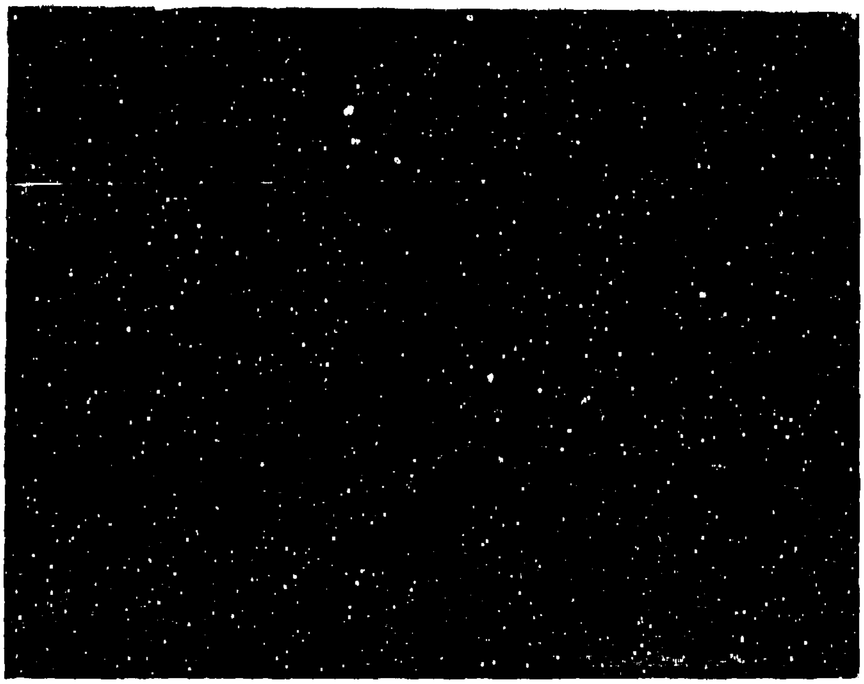


Figure 22: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled to 70% Reduction in Thickness and Annealed Four Hours at 1300°F (704°C). Specimen Etched; 400 X Magnification

Table 6: Rockwell "C" Hardness Data for MA 754 Cold Rolled to 70% Reduction in Thickness, Parallel to Extrusion Direction and Annealed Four Hours.

ANNEAL TEMP (°F)	HARDNESS - R_c
As Rolled	47
1000	48.5
1100	47.5
1200	45
1300	34.5
1400	33
1500	33
1600	33
1700	33
2400	33

Figure 23: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled to 70% Reduction in Thickness and Annealed Four Hours at 2400°F (1315°C). Specimen Etched; 680 X Magnification

2.5.2 Cold Rolling of MA 754 to 35% Reduction in Thickness

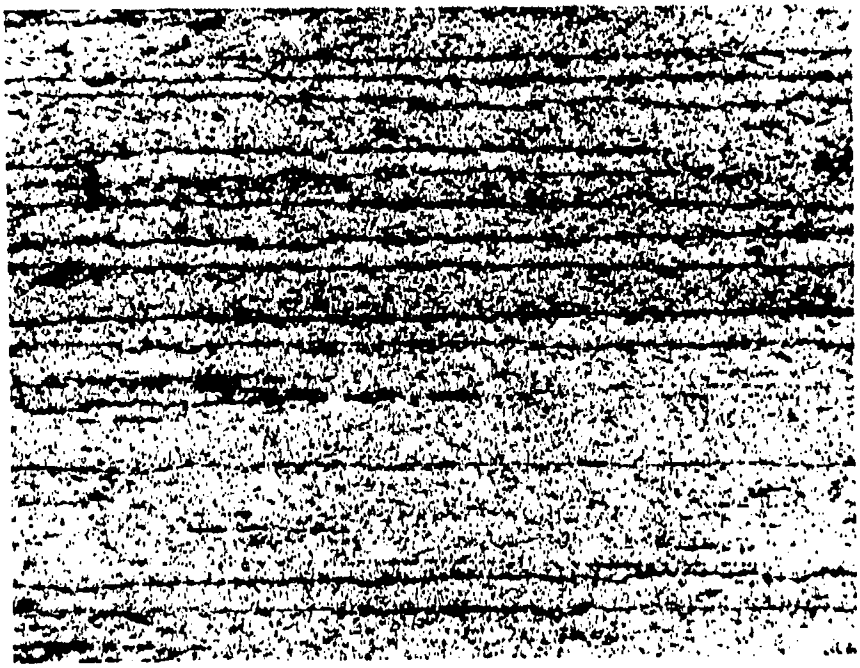
2.5.2.1 Characterization

The initial effort of ambient temperature deformation of MA 754 involved cold rolling the as-received material to a thickness reduction of some 70%, with no intermediate anneals. The second effort involved cold rolling a blank of MA 754 to 35% reduction in thickness. This blank was then subjected to annealing experiments similar to those done with MA 754 cold rolled 70% in order to determine if a reduction in the amount of stored strain energy would influence the 2400°F anneal response of cold rolled MA 754 given an intermediate anneal. Figure 24 shows the longitudinal and the transverse microstructures of the rolled material.

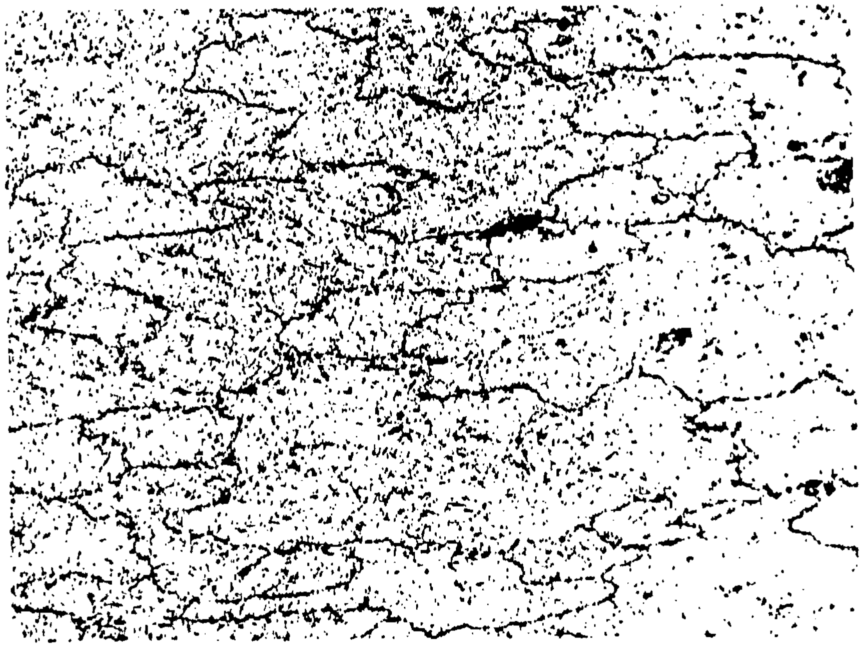
2.5.2.2 Recrystallization Response

In order to determine the recrystallization response of the cold rolled material, six separate anneals were performed. Specimens were machined from the as-rolled blank, assigned a specific temperature in the range of 1100°F to 2400°F (593°C to 1315°C) and annealed for four hours in argon.

Figure 25 is the recrystallization curve for the cold rolled material. The temperature at which recrystallization was first apparent was 1500°F (815°C), Figure 26, which resulted in a 10% drop in hardness as compared to the as-rolled hardness value. A complete list of microhardness values for each of the annealed specimens is provided in Table 7.



a. Longitudinal Section



b. Transverse Section

Figure 24: Optical Photomicrographs of a Cold Rolled Plate of MA 754; 35% Reduction in Thickness.
Specimen Etched; 60 X Magnification

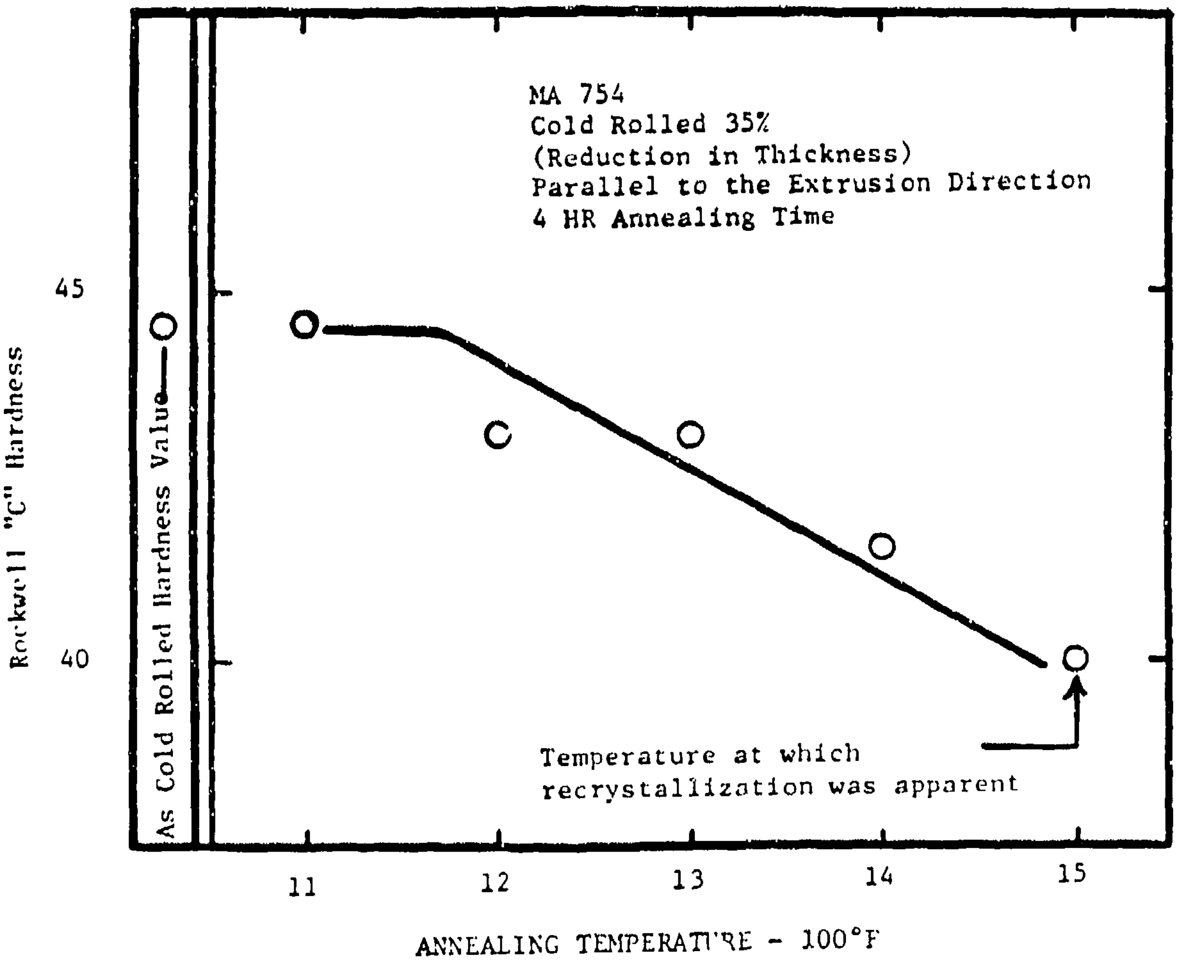


Figure 25: Recrystallization Curve for MA 754 Cold Rolled Parallel to the Extrusion Direction to 35% Reduction in Thickness.

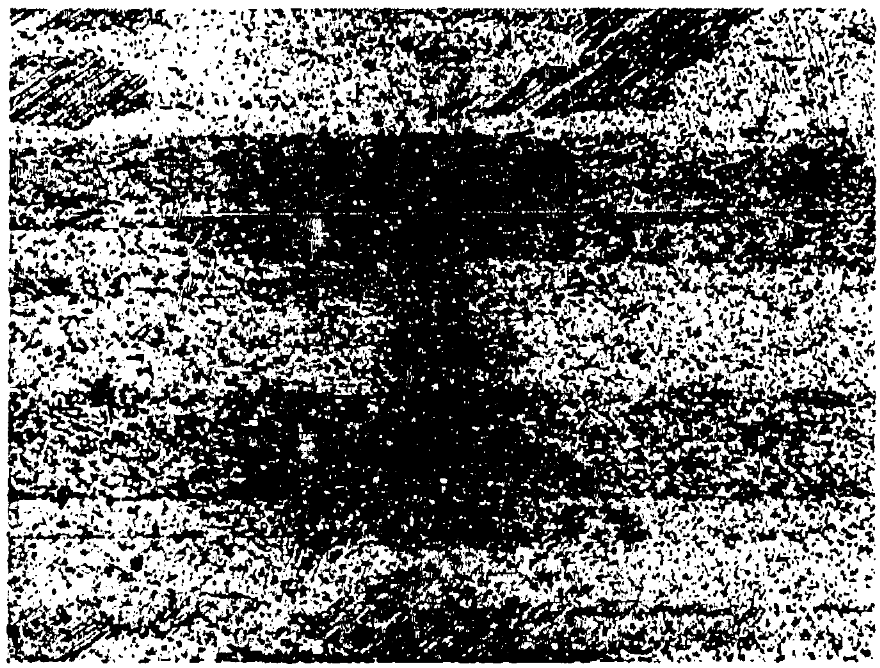


Figure 26: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled to a Reduction of 35% (in Thickness) and Annealed Four Hours at 1500°F (815°C).
Etched Condition; 160 X Magnification.
Note the beginning of recrystallization.

Table 7: Rockwell "C" Hardness Data for MA 754 Cold Rolled to 35% Reduction in Thickness, Parallel to Extrusion Direction, Annealed Four Hours

ANNEAL TEMP (°F)	HARDNESS - R_c
As Rolled	44.5
1100	44.5
1200	43
1300	43
1400	41.5
1500	40
2400	28

2.5.2.3 Intermediate Annealing Response

Three samples of MA 754 cold rolled 35% were used in this experiment:

> As Rec'd MA 754 Cold Rolled 35% + Anneal at 2400°F, four hours (in argon)
>
> As Rec'd MA 754 Cold Rolled 35% + Anneal at 1600°F, four hours + Anneal at 2400°F, four hours (in argon)
>
> As Rec'd MA 754 Cold Rolled 35% + Anneal at 1100°F, 1200°F, 1300°F, 1400°F, four hours each + Anneal at 2400°F, four hours (in argon)

Figure 27 is an optical photomicrograph revealing a microstructure common to all three samples. The following conclusions seem warranted:

1) The intermediate anneals apparently have no effect on structure, since as-rolled samples produced a similar microstructure upon annealing at 2400°F.

2) The recrystallization response of MA 754 appears to be controlled primarily by the amount of cold work, as shown in Figures 23 and 27.

2.6 Elevated Temperature Tensile Properties of Cold Worked and Annealed MA 754

In order to determine the basic strength and ductility of MA 754 cold rolled 35% and annealed at 2400°F (1315°C) for four hours in air, tensile tests were performed (in duplicate) at temperatures ranging from ambient to 2000°F (1093°C).

The specimen design used to examine the tensile properties of the cold rolled material is shown in Figure 28. Generous radii of curvature at the specimen shoulders were employed to minimize the possibility of shoulder breaks. All tests were conducted utilizing an Instron test

Figure 27: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled to 35% Reduction in Thickness and Annealed at 2400°F (1315°C) for 4 hours. Specimen Etched; 420 X Magnification.

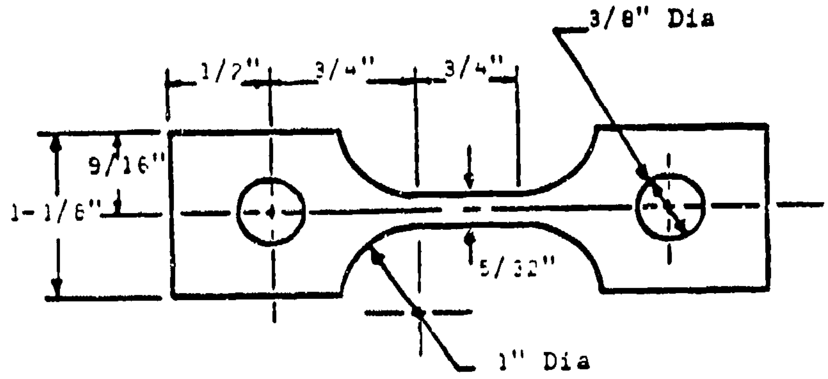


Figure 28: Design of Test Piece used for Tensile Testing of the Cold-Rolled Plus Annealed Material.

machine coupled with a Satec tube type furnace. The temperature was controlled to ± 2°F (± 1.1°C) throughout the gage section using 3 Chromel - Alumel thermocouples for temperature sensing. A cross-head speed of 0.05 in/min was employed for all tests.

Table 8 gives the results of the tensile tests of the cold rolled plus annealed material in addition to the tensile properties of the as-received alloy. These results show that the cold rolled plus annealed material had lost a certain amount of ductility, especially at the higher temperatures when compared to the as-received material. However, it is significant to note that the cold rolled plus annealed material maintains the same basic strength of the as-received alloy at all temperatures.

Microscopic examination of the cold rolled plus annealed tensile test fractured specimen revealed two primary failure modes. At temperatures below 1000°F (538°C), transgranular failure is readily apparent (Figure 29). Above 1000°F, intergranular failure is observed (Figure 30). As is the case of the as-received material, the locus of failure in the intergranular mode for the cold rolled plus annealed material is at the transverse grain boundaries.

2.7 Creep Properties of MA 754 Cold Rolled 35% and Annealed at 2400°F

All creep testing of the cold rolled plus annealed MA 754 was performed in tension using the same specimen and grip designs utilized for elevated temperature tensile testing of this material. The testing temperature and stress level for all tests conducted were 1800°F (982°C) and 21,250 psi (146.5 MPa), 0.85σF at 1800°F, respectively.

Table 8: Tensile Properties of MA 754 in Two Conditions

Condition: Cold Rolled 35% + Anneal 2400°F, 4 Hrs

°F	TENSILE STRENGTH (PSI)	.2% YIELD STRENGTH (PSI)	% ELONGATION
Ambient	138,500	88,300	13.4
1000	117,700	67,100	14.3
1400	49,400	40,300	8.5
1750	26,400	24,800	5.7
2000	17,500	15,900	2.9

Condition: As-Received

°F	TENSILE STRENGTH (PSI)	.2% YIELD STRENGTH (PSI)	% ELONGATION
Ambient	142,700	87,300	19.8
1000	113,900	65,100	19.4
1400	48,800	42,400	38.7
1750	26,800	25,000	28.8
2000	18,500	17,700	14.5

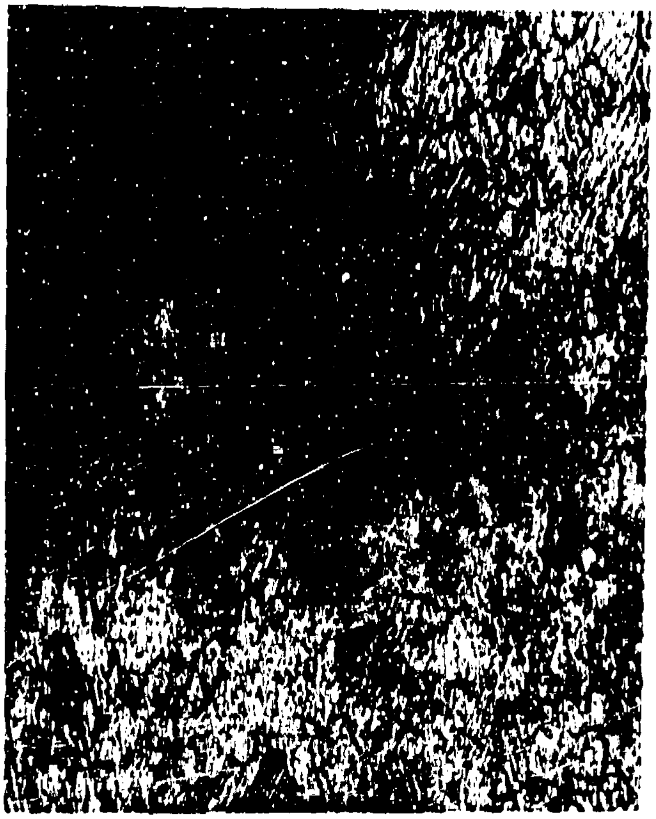


Figure 29: Optical Photomicrograph of the Mid-Section of a Longitudinal Tensile Test Piece of MA 754, Cold Rolled to 35% Reduction in Thickness and Annealed at 2400°F (1315°C) Four Hours. Test Conducted at 1000°F (538°C). Photomicrograph Shows Fracture Edge.
Specimen Etched 240 X Magnification.
Note the transgranular fracture mode.

Figure 30: Optical Photomicrograph of the Mid-Section of a Longitudinal Tensile Test Piece of MA 754, Cold Rolled to 35% Reduction in Thickness and Annealed at 2400°F (1315°C) Four Hours. Test Conducted at 2000°F (1093°C). Photomicrograph Shows Fracture Edge.
Specimen Etched 160 X Magnification.
Note intergranular fracture mode.

Table 9 shows the results of the creep tests of the cold rolled, plus annealed material, in addition to the creep properties of the as-received alloy. These results show that the cold rolled plus annealed material has lost a significant amount of the creep resistance inherent in the as-received material.

2.8 Annealing Response of MA 754 Cold Rolled To 20% Reduction in Thickness

The results of the cold work plus anneal experiments reported earlier appear to indicate that the recrystallization response of the alloy is controlled primarily by the amount of cold work (refer to Figure 23 and 27). The samples cold rolled 35% plus anneal did not recrystallize to as fine a grain size as the 70% cold-rolled samples.

In order to provide further evidence to support the observation stated above, the as-received MA 754 alloy was cold rolled to 20% reduction in thickness and annealed at 2400°F (1315°C) for four hours in air. Microscopic examination reveals that some sections have a recrystallized structure similar to that shown in Figure 27 (35% reduction plus anneal), while other areas appear unchanged from the original 20% cold worked structure (Figures 31 and 32).

In order to apply the microscopic evidence given above in the attempt to recover the loss in ductility described earlier, the as-received MA 754 was cold rolled 20% and annealed at 2400°F (1315°C) for four hours in air and tested in tension at 2000°F (1093°C). The results (Table 10) indicate a substantial recovery of tensile ductility.

Table 9: Creep Properties of MA 754 at 1800°F (982°C) and 21.25 KSI (146.5 MPa) in Two Conditions

CONDITION	MINIMUM CREEP RATE $\dot{\epsilon}$/sec	RUPTURE TIME (HRS)	RUPTURE ELONGATION (%)
As Rec'd	1.55×10^{-7}	70	10.1
Cold Rolled 35% + Anneal at 2400°F, 4 hrs.	1.16×10^{-4}	.06	9.3

Figure 31: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled 20% Reduction in Thickness, and Annealed Four Hours at 2400°F (1315°C).
Specimen Etched 160 X Magnification.
Note that the microstructure is similar to that found in specimens cold rolled 35% and annealed at 2400°F (1315°C).

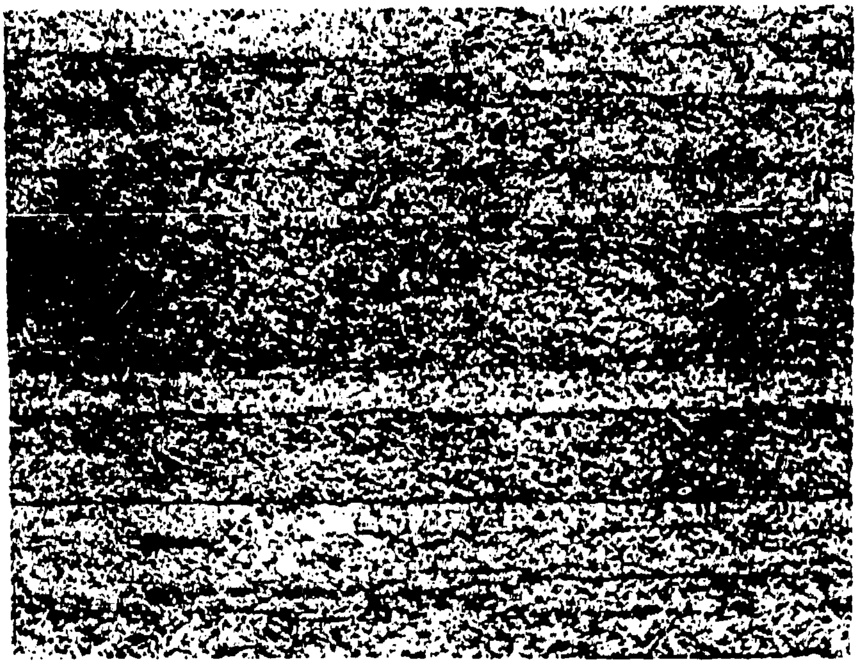


Figure 32: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled 20% Reduction in Thickness, and Annealed Four Hours at 2400°F (1315°C).
Specimen Etched 160 X Magnification
Note that the micrograph shows an absence of recrystallization.

Table 10: Tensile Properties of MA 754 in Three Conditions at 2000°F (1093°C)

CONDITION	TENSILE STRENGTH (PSI)	.2% YIELD STRENGTH (PSI)	% ELONGATION
As Received	18,500	17,700	14.5
Cold Rolled 35% + Anneal At 2400°F, 4 Hrs.	17,500	15,900	2.9
Cold Rolled 20% + Anneal At 2400°F, 4 Hrs.	20,600	19,200	10.0

2.9 Effects of Cyclic Cold Working and Annealing MA 754

In the attempt to utilize the beneficial effects of reducing the amount of cold work fully while retaining useful reductions, the cold rolling plus anneal schedule was revised into one involving step-wise cold working and annealing:

STEP 1: Cold Roll as Rec'd MA 754 x%

STEP 2: Anneal Material in (1) at 2400°F, four hours (in air)

STEP 3: Cold Roll the Annealed Material in (2) x%

STEP 4: Anneal Material in (3) at 2400°F, four hours (in air)

Subsequent steps, as warranted, according to above.

An intregal part of this schedule involved determining the maximum deformation ("x%") that can be applied during each rolling cycle without recrystallization occurring upon subsequent annealing. This was accomplished by cold-rolling a wedge-shaped blank 3.7" long x 1.3" wide x 0.35" thick at the large end (94.0 mm x 33.0 mm x 8.9 mm), with a 5.4° included angle. The resulting reduction in thickness within the piece varied from 0 to 35%. The blank was then annealed at 2400°F, in air, for four hours. Because of the cold work gradient, part of the bar (that with the higher amounts of cold work) recrystallized while the part with subcritical amounts of cold work did not. From metallographic examination of the annealed wedge the maximum reduction, "x", was determined for the processing schedule.

The results of this experiment indicate that cold rolling MA 754 to 10% reduction in thickness, plus annealing at 2400°F, produces a small amount of recrystallized structure (Figure 23). Therefore,

the maximum reduction, "x", was taken to be 5%.

Figures 34 through 37 are optical photomicrographs of MA 754 cold rolled and annealed to three different total reductions according to the revised schedule, along with as-received MA 754 annealed at 2400°F for four hours in air. It appears that utilization of the revised cold rolling plus annealing schedule permits total reductions of at least 40% while still maintaining the favorable grain morphology of as-received MA 754.

In an attempt to characterize the material that was cold rolled plus annealed according to the revised schedule, Rockwell hardness measurements were taken using samples with total reductions ranging from 0 to 40% (Table 11 and Figure 38). (For comparison purposes, the Rockwell hardness measurements of MA 754 cold rolled 20% and 35% in one step with no anneals are plotted on the graph). The results of these experiments serve to warrant two rather unexpected observations:

1) MA 754 cold rolled and annealed according to the revised schedule has recrystallized yet maintained a favorable grain morphology.

2) The hardness of MA 754 cold rolled and annealed according to the revised schedule does not appear to vary significantly with increasing reduction. This observation is consistent with the structures observed in Figures 34 through 37.

Figure 33: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled 10% Reduction in Thickness and Annealed Four Hours at 2400°F (1315°C).
Etched Condition 200 X Magnification
Note evidence of recrystallization.

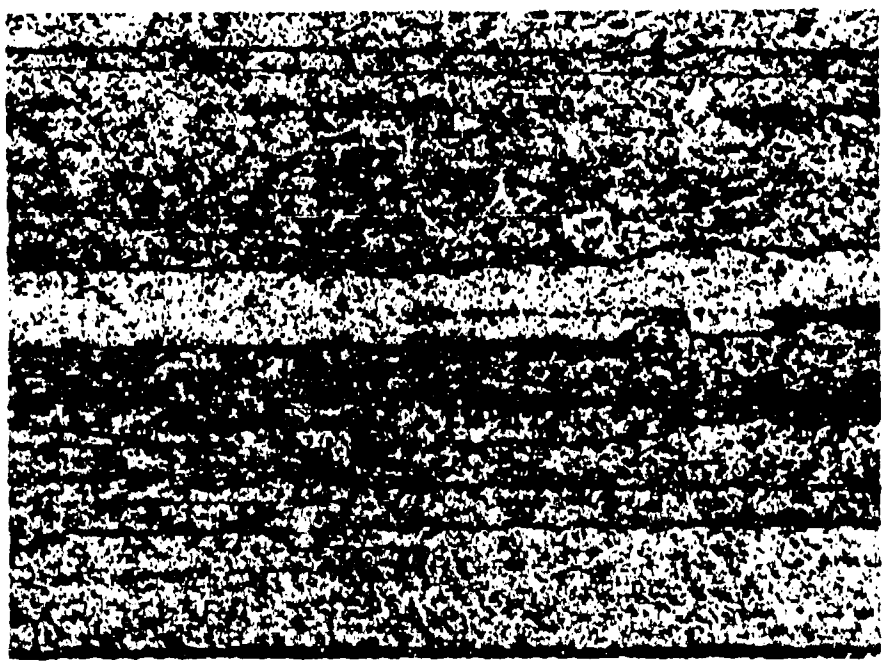


Figure 34: Optical Photomicrograph of a Longitudinal Section of MA 754, As-Received and Annealed Four Hours at 2400°F (1315°C).
Etched Condition 200 X Magnification

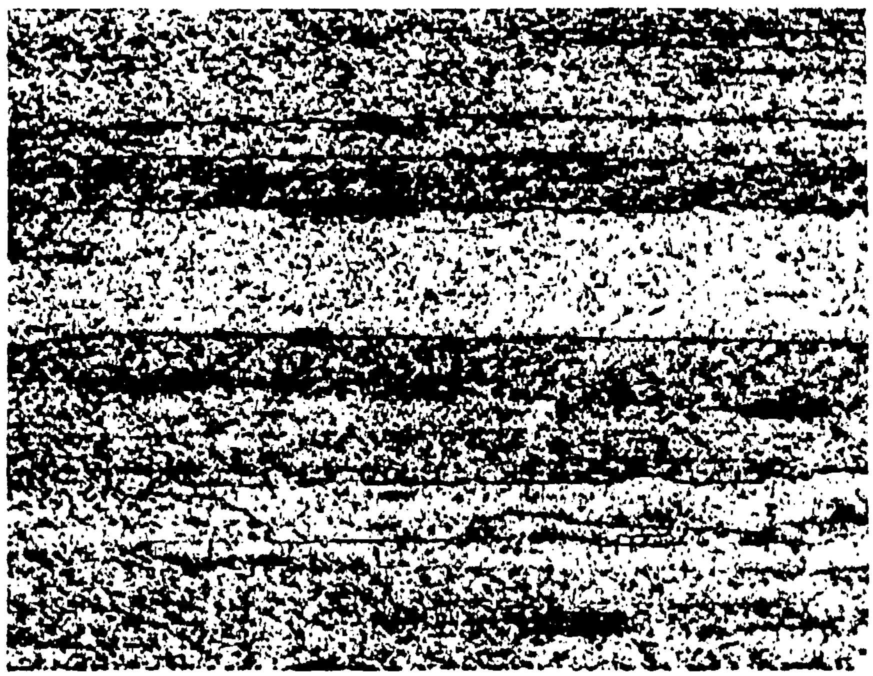


Figure 35: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled 15% Reduction in Thickness and Annealed According to the Revised, Stepwise Schedule of Cold Rolling and Annealing.
Etched Condition 100 X Magnification

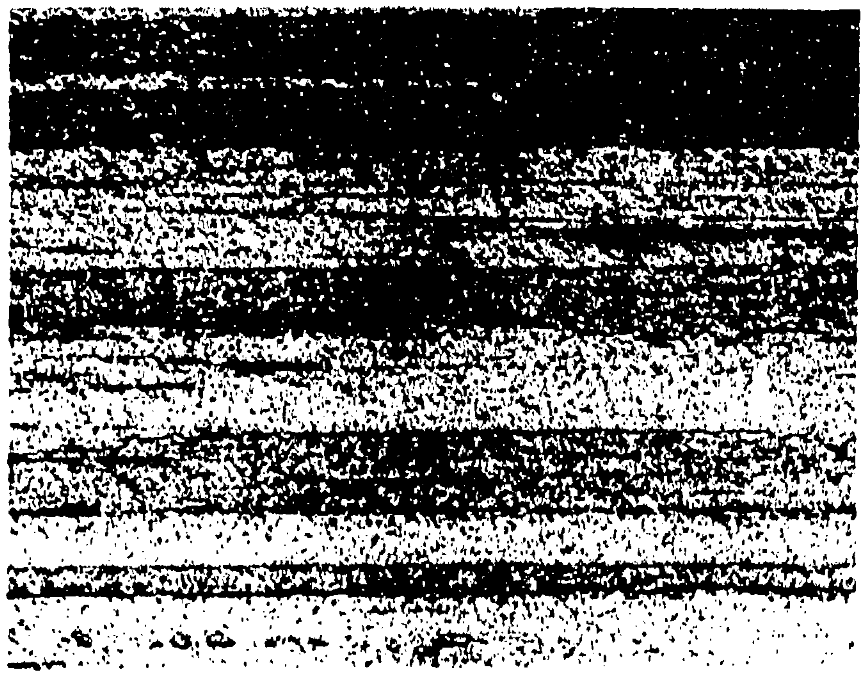


Figure 36: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled 22% Reduction in Thickness and Annealed According to the Revised, Stepwise Schedule of Cold Rolling and Annealing.
Etched Condition 100 X Magnification

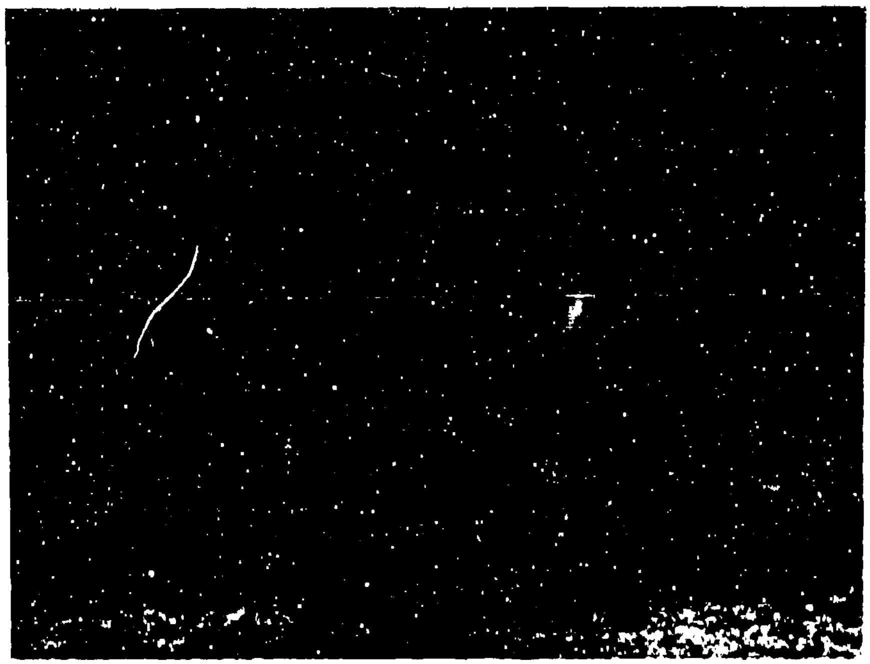


Figure 37: Optical Photomicrograph of a Longitudinal Section of MA 754 Cold Rolled 40% Reduction in Thickness and Annealed According to the Revised, Stepwise Schedule of Cold Rolling and Annealing. Etched Condition 100 X Magnification

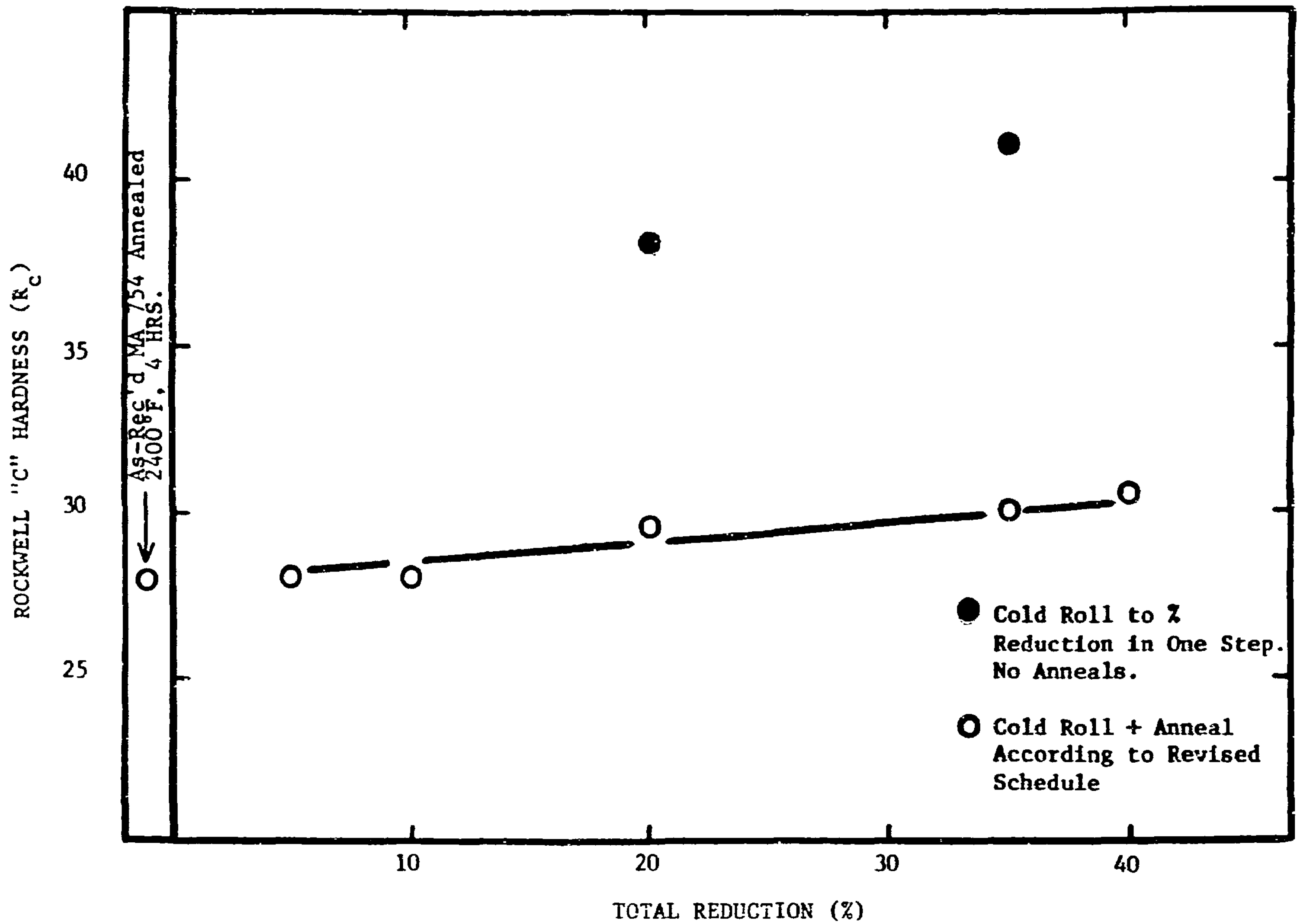


Figure 38: Rockwell "C" Hardness Versus Total Reduction in Thickness by Cold Rolling Under Two Different Conditions.

Table 11: Rockwell "C" Hardness Data for MA 754 Cold Rolled Under Two Different Conditions

Cold Roll + Anneal According to Revised Schedule

% Reduction	Hardness - R_c
0	28
5	28.5
10	28.1
20	29.5
35	30.0
40	30.7

Cold Roll in One Step, No Anneals

% Reduction	Hardness - R_c
20	38
35	41

3.0 CONCLUSIONS

On the basis of the work completed to date, the following conclusions seem warranted:

1. The as-received MA 754 exhibits excellent high temperature tensile and creep properties. This is consistent with the favorable grain morphology observed.

2. Rupture during high temperature tensile and creep deformation in MA 754 is intergranular and occurs as a result of cracking at transverse grain boundaries.

3. The stress dependence of MA 754 in creep is very high at both temperatures studied and appears to be dependent upon temperature. However, the variation in the stress exponent with temperature is consistent with other observations for ODS materials.

4. It appears that MA 754 is very amenable to cold rolling. Reductions of at least 70% were obtained with no evidence of edge or side cracking of the rolling blanks.

5. Results of various cold rolling plus anneal experiments appear to indicate that the excellent grain morphology obtained by cold rolling cannot be stabilized through the use of intermediate anneals. However, it was noted that the severity of recrystallization is controlled primarily by the amount of the cold work.

6. Through application of the above observation, a revised cold rolling plus anneal schedule was devised which was successful in obtaining total reductions on the order of at least 40% while at the same time maintaining the favorable grain morphology found in the as-received material.

4.0 RECOMMENDATIONS

Since the data developed in this study indicates that the ODS alloy MA 754 can be formed to nearer net-shape than previously thought without recrystallization to small grain size at the elevated use temperatures, it is recommended that the investigation be pursued to evaluate the full potential of this finding. Specifically, it is suggested that the basic mechanism responsible for the retention of the large grain size after the stepwise cold work and anneal be determined, and that the effect be maximized. In this latter respect, it is recommended that the parameters which affect the large grain size retention phenomenon, such as stress state of the cold deformation, the temperature of the cold deformation, etc., be investigated for optimization purposes. Finally, it is recommended that the continued study include the generation of the standard creep and creep-rupture data required for use in engineering design applications.

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HOWMET CORP.
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Bloomfield, CT 06002 1

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SPECIAL METALS CORPORATION
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